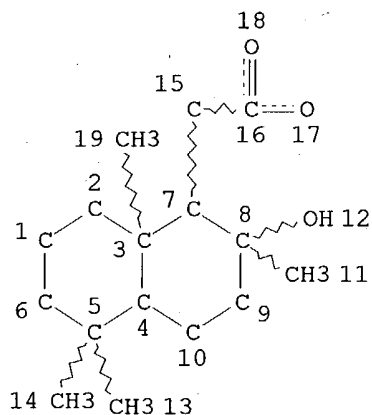


Reyes, H.  
10/820709

10/820709

(FILE 'REGISTRY' ENTERED AT 12:48:13 ON 28 OCT 2004)

L1 STR



NODE ATTRIBUTES:  
DEFAULT MLEVEL IS ATOM  
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:  
RSPEC I  
NUMBER OF NODES IS 19

STEREO ATTRIBUTES: NONE  
L3 16 SEA FILE=REGISTRY SSS FUL L1

100.0% PROCESSED 522 ITERATIONS  
SEARCH TIME: 00.00.01

16 ANSWERS

(FILE 'CAPLUS' ENTERED AT 12:50:36 ON 28 OCT 2004)

L4 29 S L3

L4 ANSWER 1 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2004:120806 CAPLUS

DOCUMENT NUMBER: 140:164046

TITLE: A process for the optical resolution of a precursor of sclareolide

INVENTOR(S): Huboux, Alexandre

PATENT ASSIGNEE(S): Firmenich SA, Switz.

SOURCE: PCT Int. Appl., 15 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004013069	A1	20040212	WO 2003-IB2933	20030724
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,				

Searcher : Shears 571-272-2528

10/820709

LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM,  
PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN,  
TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY,  
KG, KZ, MD, RU  
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG,  
CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC,  
NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ,  
GW, ML, MR, NE, SN, TD, TG

US 2004192960

A1

20040930

US 2004-820709

20040409

PRIORITY APPLN. INFO.:

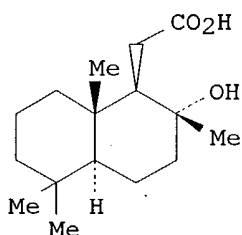
WO 2002-IB3055

A 20020731

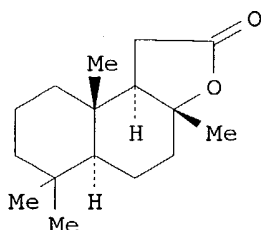
OTHER SOURCE(S):

CASREACT 140:164046

GI



I



II

AB The present invention relates to the field of organic synthesis and more particularly to a new process for the optical resolution of a precursor of sclareolide. Said process is characterized by the reaction of [(1R,2R,4aS,8aS)-2-hydroxy-2,5,5,8a-tetramethyldecahydronaphthalen-1-yl]acetic acid (I), or an alkaline salt thereof, with an enantiomer of the 2-(methylamino)-1-phenyl-1-propanol, or an ammonium salt thereof resp., which is used as resolving agent. Thus, I was treated with (1R,2R)-pseudoephedrine in THF to form the diastereomeric salt of (1R,2R,4aS,8aS)-I with (1R,2R)-pseudoephedrine. The diastereomeric salt was treated with 10% aqueous H<sub>2</sub>SO<sub>4</sub> in toluene and the toluene phase containing

(1R,2R,4aS,8aS)-I was subsequently treated with acetic acid to give (+)-sclareolide (II) in 91% yield and >98% ee.

IT **151123-71-6P**, [(1R,2R,4aS,8aS)-2-hydroxy-2,5,5,8a-tetramethyldecahydronaphthalen-1-yl]acetic acid **654076-05-8P**

RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

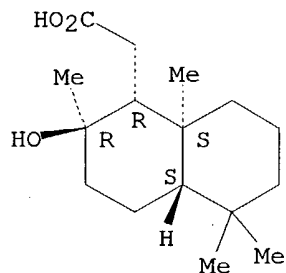
(process for the optical resolution of an open-acid precursor of sclareolide and asym. synthesis of sclareolide)

RN 151123-71-6 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.

10/820709



RN 654076-05-8 CAPLUS

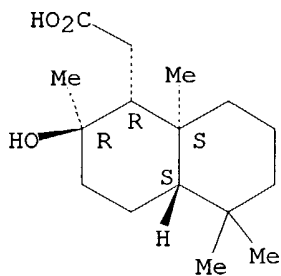
CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-,  
(1R,2R,4aS,8aS)-, compd. with (αR)-α-[(1R)-1-(methylamino)ethyl]benzenemethanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 13456-36-5

CMF C16 H28 O3

Absolute stereochemistry.

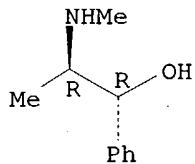


CM 2

CRN 321-97-1

CMF C10 H15 N O

Absolute stereochemistry. Rotation (-).



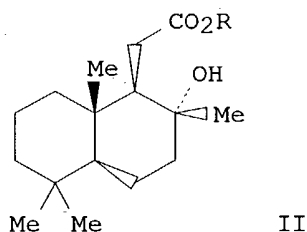
L4 ANSWER 2 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 2002:975640 CAPLUS

Searcher : Shears 571-272-2528

10/820709

DOCUMENT NUMBER: 138:39432  
TITLE: One-step preparation of sclareolide from sclareol  
INVENTOR(S): Adachi, Kenichiro; Matsuda, Hiroyuki  
PATENT ASSIGNEE(S): Takasago Perfumery Co., Ltd., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.  
CODEN: JKXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2002371031	A2	20021226	JP 2001-183872	20010618
PRIORITY APPLN. INFO.:			JP 2001-183872	20010618
OTHER SOURCE(S):	CASREACT 138:39432			
GI				



AB Sclareolide (I) or its hydrolyzed ring-opened compds. II (R = H, alkali metal, alkaline earth metal), useful as intermediates for fragrant materials, are prepared by oxidation of sclareol (III) in the presence of alkali substances, Ru catalysts, hypochlorite salts, phase-transfer catalysts, and cyclic hydrocarbon solvents. Thus, III was oxidized with aqueous NaOCl in

the presence of RuCl<sub>3</sub>, Bu<sub>4</sub>N+HSO<sub>4</sub><sup>-</sup>, aqueous NaOH, and MePh at 35-45° for 2.5 h and cyclized to give 75.0% I.

IT **13456-36-5P 478977-46-7P**

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

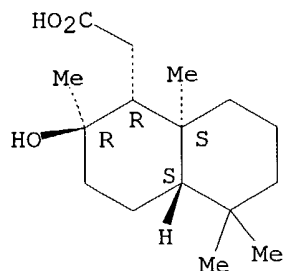
(one-step preparation of sclareolide as intermediate for fragrant substances from sclareol)

RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

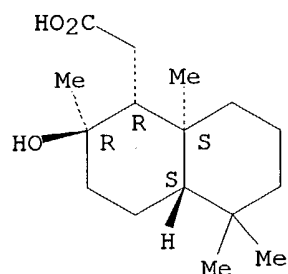
10/820709



RN 478977-46-7 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, monosodium salt, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



● Na

L4 ANSWER 3 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2001:782242 CAPLUS

DOCUMENT NUMBER: 136:340845

TITLE: Preparation of sclareolide by sclareol ozonolysis. *mw*

AUTHOR(S): Fekih, A.; Habbachi, F.

CORPORATE SOURCE: Laboratoire de Chimie, Departmes Fondamentales et Mixtes, Faculte de Medecine Dentaire, Monastir, 5000, Tunisia

SOURCE: Journal de la Societe Chimique de Tunisie (2001), 4(9), 909-914

CODEN: JSCTDP; ISSN: 0253-1208

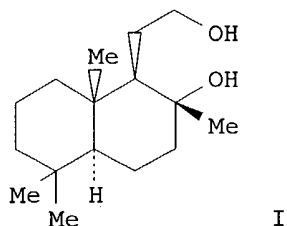
PUBLISHER: Societe Chimique de Tunisie

DOCUMENT TYPE: Journal

LANGUAGE: French

GI

10/820709



I

AB Sclareolide was obtained in high yield (> 96%) in a one pot reaction from sclareol by ozonolysis and treatment in situ by H<sub>2</sub>O<sub>2</sub>/NaOH/CH<sub>3</sub>CO<sub>3</sub>H. This com. interesting product gave Ambrox in very good yield (total yield from sclareol > 85%) by reduction and subsequent cyclization of the intermediary bicyclohomofarnesane 8α-12diol (I).

IT 13456-36-5P

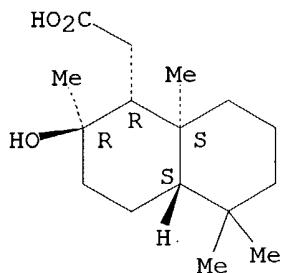
RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of sclareolide by sclareol ozonolysis and three step synthesis of Ambrox)

RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 4 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2000:814 CAPLUS

DOCUMENT NUMBER: 132:207466

TITLE: Phenylglycine Methyl Ester, a Useful Tool for Absolute Configuration Determination of Various Chiral Carboxylic Acids

AUTHOR(S): Yabuuchi, Tetsuya; Kusumi, Takenori

CORPORATE SOURCE: Faculty of Pharmaceutical Sciences, Tokushima University, Tokushima, 770-8505, Japan

SOURCE: Journal of Organic Chemistry (2000), 65(2), 397-404

CODEN: JOCEAH; ISSN: 0022-3263

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

Searcher : Shears 571-272-2528

10/820709

LANGUAGE: English

AB A new chiral anisotropic reagent, phenylglycine Me ester (PGME), developed for the elucidation of the absolute configuration of chiral  $\alpha,\alpha$ -disubstituted acetic acids, has turned out to be applicable to other substituted carboxylic acids, such as chiral  $\alpha$ -hydroxy-,  $\alpha$ -alkoxy-, and  $\alpha$ -acyloxy  $\alpha,\alpha$ -disubstituted acetic acids, as well as to chiral  $\beta,\beta$ -disubstituted propionic acids. Because a carboxylic moiety is convertible from other functional groups, e.g., ozonolysis of an olefin and oxidative cleavage of a glycol, the present findings can expand the utility of the PGME method to the absolute configuration determination of various types of organic compds., even those which initially lack oxygen functions. Several examples of the combination of chemical reactions and the PGME method are described.

IT 13456-36-5P

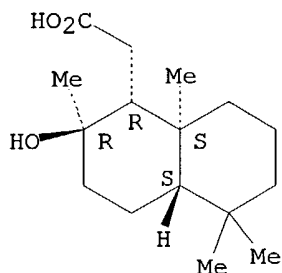
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(determination of absolute configuration of chiral carboxylic acids using phenylglycine Me ester as anisotropic reagent)

RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 5 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1999:690945 CAPLUS

DOCUMENT NUMBER: 131:307085

TITLE: Antifungal agents

INVENTOR(S): Nozoe, Shigeo; Masuda, Jun-ichi; Takahashi, Akira; Kanou, Muneaki; Tanaka, Ken-ichi; Wakayama, Toshiyuki; Koike, Nobuaki; Uchida, Takayoshi; Nagata, Toshiyuki; Segawa, Toshiaki; Tanaka, Sanae

PATENT ASSIGNEE(S): Toa Gosei Co., Ltd., Japan

SOURCE: PCT Int. Appl., 203 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

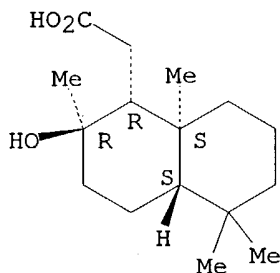
Searcher : Shears 571-272-2528

10/820709

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9953911	A1	19991028	WO 1999-JP1998	19990414
W: JP, US				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
JP 2002179564	A2	20020626	JP 1998-120011	19980414
PRIORITY APPLN. INFO.:			JP 1998-120011	A 19980414
AB Antifungal agents containing as the active ingredient compds. having a hydronaphthalene ring structure, in particular, albicanol in the hydronaphthalene ring structure moiety and sclareol, sclareolide, manool, labdanolic acid etc. being similar in structure thereto each optionally having various substituents. These antifungal agents are efficacious against fungi inducing opportunistic infection.				
IT <b>13456-36-5P</b>				
RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); RCT (Reactant); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation); RACT (Reactant or reagent) (hydronaphthalene analogs as antifungal agents for treatment of opportunistic infection)				
RN	13456-36-5 CAPLUS			
CN	1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)			

Absolute stereochemistry.



REFERENCE COUNT: 8 THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 6 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1998:805222 CAPLUS

DOCUMENT NUMBER: 130:153814

TITLE: Resolution of sclareolide as a key intermediate for the synthesis of Ambrox

AUTHOR(S): Koga, Tsukasa; Aoki, Yoshio; Hirose, Takuji; Nohira, Hiroyuki

CORPORATE SOURCE: Department of Applied Chemistry, Faculty of Engineering, Saitama University, Urawa, Saitama, 338-8570, Japan

SOURCE: Tetrahedron: Asymmetry (1998), 9(21), 3819-3823

CODEN: TASYE3; ISSN: 0957-4166

PUBLISHER: Elsevier Science Ltd.

Searcher : Shears 571-272-2528



10/820709

DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 130:153814

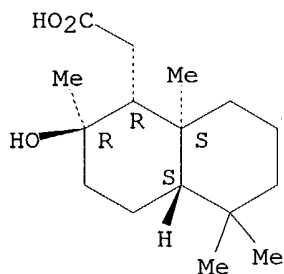
AB Sclareolide was efficiently resolved by a diastereomeric salt formation method using homochiral erythro-2-amino-1,2-diphenylethanol (ADPE) as a resolving agent. Synthesis of the enantiomerically pure Ambrox was accomplished via the resolved (+)-sclareolide.

IT 220202-44-8P 220202-45-9P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(resolution of sclareolide as a key intermediate for the synthesis of Ambrox)

RN 220202-44-8 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, monosodium salt, (1R,2R,4aS,8aS)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



● Na

RN 220202-45-9 CAPLUS

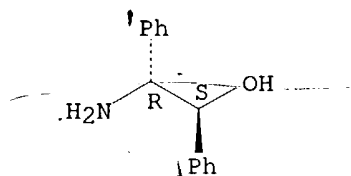
CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)-, compd. with (αS,βR)-β-amino-α-phenylbenzeneethanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 23364-44-5

CMF C14 H15 N O

Absolute stereochemistry. Rotation (+).



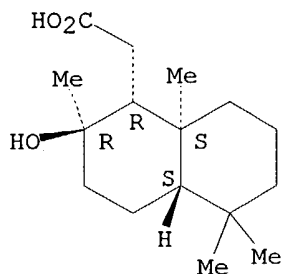
CM 2

Searcher : Shears 571-272-2528

10/820709

CRN 13456-36-5  
CMF C16 H28 O3

Absolute stereochemistry.



REFERENCE COUNT: 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 7 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1998:488530 CAPLUS

DOCUMENT NUMBER: 129:159295

TITLE: New types of potentially antimalarial agents.  
Epidioxy-substituted norditerpene and  
norsesterterpenes from the marine sponge *Diacarnus levii*

AUTHOR(S): D'Ambrosio, Michele; Guerriero, Antonio; Deharo, Eric;  
Debitus, Cecile; Munoz, Victoria; Pietra, Francesco

CORPORATE SOURCE: Laboratorio Chimica Bioorganica, Universita Trento,  
Trento, I-38050, Italy

SOURCE: Helvetica Chimica Acta (1998), 81(7), 1285-1292

CODEN: HCACAV; ISSN: 0018-019X

PUBLISHER: Verlag Helvetica Chimica Acta AG

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Natural free carboxylic acids from the hadromerid sponge *Diacarnus levii* were esterified to yield the new cyclic norditerpene peroxides ent-muqubilin benzyl ester (I), diacarnate B Me ester (II), and deoxydiacarnate B benzyl ester (III) which were isolated following extensive chromatog. The relative configuration of the peroxide/ $\alpha$ -methylacetate moiety of I-III was directly determined from their NMR. The absolute configuration of the peroxide/ $\alpha$ -methylacetate moiety was deduced from comparative <sup>1</sup>H-NMR of the corresponding (S)- and (R)-phenylglycine Me ester derivs. The absolute configuration at the carbobicyclic moiety of II and of III is identical, as established by chemical interconversion. Compds. II and III belong to the normal labdane series according to empirical CD rules, applied either directly to II or to a parent (+)-sclareolide-derived enone. In contrast, molar rotation additivity rules suggest the ent-labdane configuration for II and III. Me diacarnate A, me 3-epinuapapuanate, 2-epimukubulin benzyl ester, II, and III proved active in vitro against the malaria parasite *Plasmodium falciparum*. Especially the previously isolated Me 3-epinuapapuanate was active

against a chloroquine-resistant strain with a good security index.

Searcher : Shears 571-272-2528

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IT 137960-53-3P

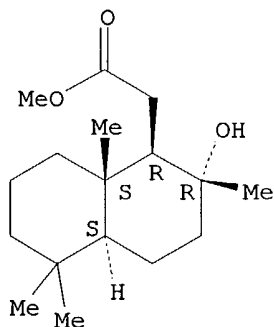
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)

(absolute configuration of di- and sesterterpenes from Diacarnus)

RN 137960-53-3 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-,  
methyl ester, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L4 ANSWER 8 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1994:631087 CAPLUS

DOCUMENT NUMBER: 121:231087

TITLE: Synthesis of norambreinolide from (+)-cis-abienol

AUTHOR(S): Barrero, Alejandro F.; Sanchez, Juan F.;  
Alvarez-Manzaneda, Enrique J.; Altarejos, Joaquin;  
Munoz, Manuel; Haidour, Ali

CORPORATE SOURCE: Dep. Quim. Org., Fac. Cienc., Granada, 18071, Spain

SOURCE: Tetrahedron (1994), 50(22), 6653-62

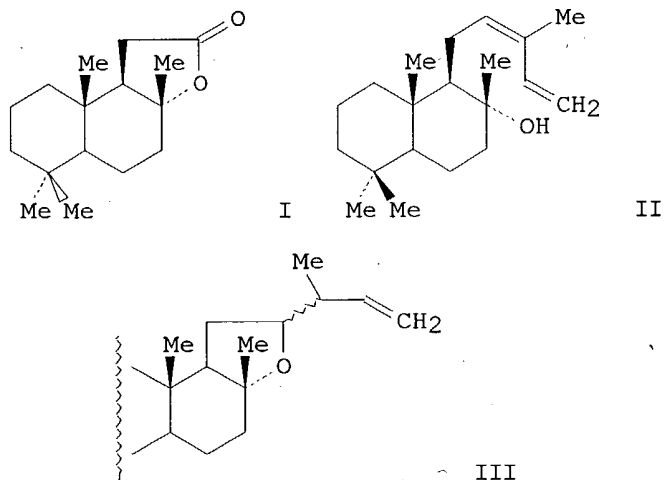
CODEN: TETRAB; ISSN: 0040-4020

DOCUMENT TYPE: Journal

LANGUAGE: English

GI

10/820709



AB The synthesis of norambreinolide (I) from cis-abienol (II) was carried out by direct treatment with OsO<sub>4</sub>-NaIO<sub>4</sub> or RuO<sub>4</sub>-NaIO<sub>4</sub>. Oxymercuration-demercuration of I led to mixture of 8,12-epoxylabdanes, e.g. III, which was also converted into norambreinolide by treatment with RuO<sub>4</sub>-NaIO<sub>4</sub>. Mechanisms for the formation of the epoxy derivs. are discussed.

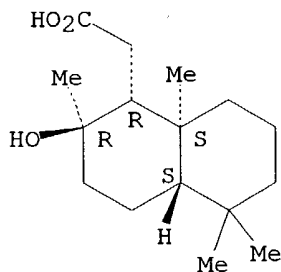
IT 13456-36-5P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation and acid-catalyzed cyclization of)

RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L4 ANSWER 9 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1994:192054 CAPLUS

DOCUMENT NUMBER: 120:192054

TITLE: Process for producing sclareolide

INVENTOR(S): Schneider, Markus; Stalberg, Theo; Gerke, Thomas

PATENT ASSIGNEE(S): Henkel K.-G.a.A., Germany

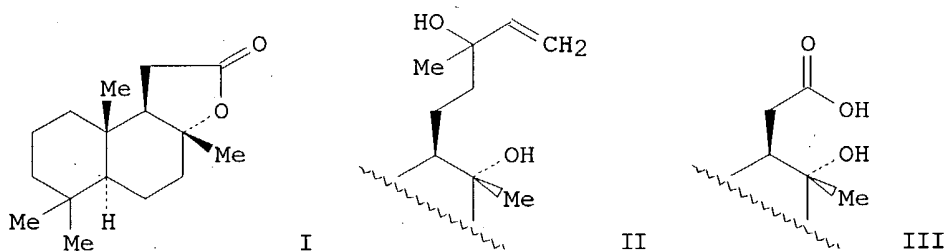
SOURCE: PCT Int. Appl., 16 pp.

Searcher : Shears 571-272-2528

10/820709

DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9321174	A1	19931028	WO 1993-EP874	19930408
W: JP, US				
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
EP 636126	A1	19950201	EP 1993-909365	19930408
EP 636126	B1	19970108		
R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, NL				
JP 07505405	T2	19950615	JP 1993-517964	19930408
JP 3213002	B2	20010925		
AT 147382	E	19970115	AT 1993-909365	19930408
ES 2095640	T3	19970216	ES 1993-909365	19930408
US 5525728	A	19960611	US 1994-318790	19941017
PRIORITY APPLN. INFO.:			DE 1992-4212731	A 19920416
			WO 1993-EP874	W 19930408
OTHER SOURCE(S):	CASREACT 120:192054			
GI				



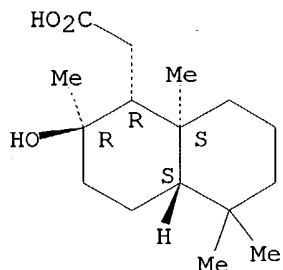
AB Sclareolide (I), an important precursor of the perfume ambroxane, is prepared by a new method on a large scale with short reaction times. The method involves oxidation of sclareol (II) or abienol in an aqueous medium in the absence of an organic solvent, using 10.5-25 mol equiv oxidizing agent in the presence of a Ru catalyst and an emulsifier, followed by treatment of the crude product in one of two ways. In the 1st, the crude is treated with a base (reacts with impurities) to give a salt of hydroxy acid III, which is cyclized in an acid medium to give I. In the 2nd method, heating of the crude to high temperature and subsequent or simultaneous distillation gives I directly. Three examples describe oxidns. of II, using RuCl<sub>3</sub> as catalyst, aqueous NaOCl as oxidant, and Dehydol TA 20 (ethoxylated tallow fatty alc.) or its combination with Disponil SMO 120 (ethoxylated sorbitan monooleate) as emulsifier. Treatment of the acidified oxidation product (as solution in PhMe) with 50% NaOH and Bu<sub>4</sub>NCl at 60-65°, acidification and extraction into

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PhMe, and reflux with removal of H<sub>2</sub>O gave 72.6-75% I, whereas direct distillation of the acidified oxidation product at 150° and 0.01 mbar gave 78% I.

IT **13456-36-5DP**, 8 $\alpha$ -Hydroxy-11-carboxy-12,13,14,15,16-pentanolabthane, salts  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and cyclization of)  
 RN 13456-36-5 CAPLUS  
 CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

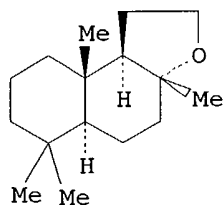


L4 ANSWER 10 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 1994:54746 CAPLUS  
 DOCUMENT NUMBER: 120:54746  
 TITLE: Process for producing L-ambrox  
 INVENTOR(S): Asanuma, Goro; Tamai, Yoshin  
 PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan  
 SOURCE: Eur. Pat. Appl., 18 pp.  
 CODEN: EPXXDW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 550889	A1	19930714	EP 1992-121945	19921223
EP 550889	B1	19960918		
R: BE, CH, DE, FR, GB, IT, LI, NL				
JP 05186388	A2	19930727	JP 1991-358777	19911229
JP 3028874	B2	20000404		
JP 05186452	A2	19930727	JP 1991-358778	19911229
US 5290955	A	19940301	US 1992-995978	19921223
US 5347048	A	19940913	US 1993-116605	19930907
PRIORITY APPLN. INFO.:			JP 1991-358777	A 19911229
			JP 1991-358778	A 19911229
			US 1992-995978	A3 19921223

OTHER SOURCE(S): CASREACT 120:54746  
 GI

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I

AB Title compound (I) a known perfumery substance is produced with a high optical purity, at a low cost and in an industrial scale by subjecting (-)-2,5,5,8a-tetramethyl-1-(carboxymethyl)-2-hydroxydecalin (II) to lactonization by dehydration, reducing the lactonized compound to (-)-2,5,5,8a-tetramethyl-1-(hydroxyethyl)-2-hydroxydecalin (III) followed by dehydrative cyclization to give I.  $\beta$ -Ionone was reduced to dihydro- $\beta$ -ionone which in 6 steps was converted to II. II was lactonized and reduced to III followed by dehydrative cyclization with p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>Cl to I.

IT 151239-43-9P 151239-44-0P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation and decomposition of)

RN 151239-43-9 CAPLUS

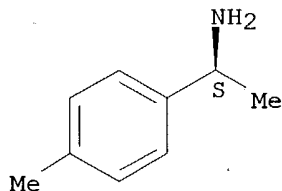
CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, [1R-(1 $\alpha$ ,2 $\beta$ ,4 $\alpha$ ,8 $\alpha$ )]-, compd. with (S)- $\alpha$ ,4-dimethylbenzenemethanamine (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 27298-98-2

CMF C9 H13 N

Absolute stereochemistry. Rotation (-).



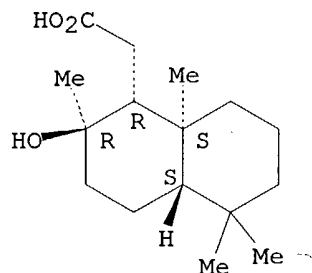
CM 2

CRN 13456-36-5

CMF C16 H28 O3

Absolute stereochemistry.

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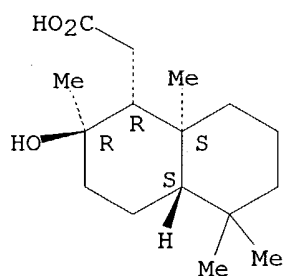
RN 151239-44-0 CAPLUS  
CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-,  
[1R-(1α,2β,4aβ,8aα)]-, compd. with  
(S)-α-methyl-1-naphthalenemethanamine (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 13456-36-5

CMF C16 H28 O3

Absolute stereochemistry.

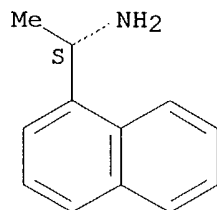


CM 2

CRN 10420-89-0

CMF C12 H13 N

Absolute stereochemistry. Rotation (-).



IT 13456-36-5P

Searcher : Shears 571-272-2528



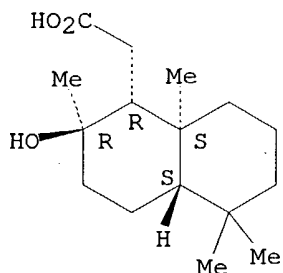
10/820709

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)  
(preparation and lactonization of)

RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-,  
(1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



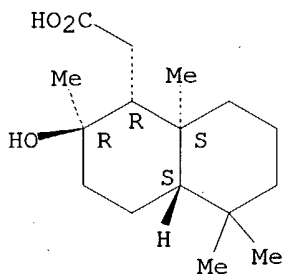
IT 151123-71-6P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 151123-71-6 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-,  
(1R,2R,4aS,8aS)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L4 ANSWER 11 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1994:8779 CAPLUS

DOCUMENT NUMBER: 120:8779

TITLE: Preparation of (±)-norambreinolide from  
β-ionone

INVENTOR(S): Asanuma, Goro; Tamai, Hironobu

PATENT ASSIGNEE(S): Kuraray Co, Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 10 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

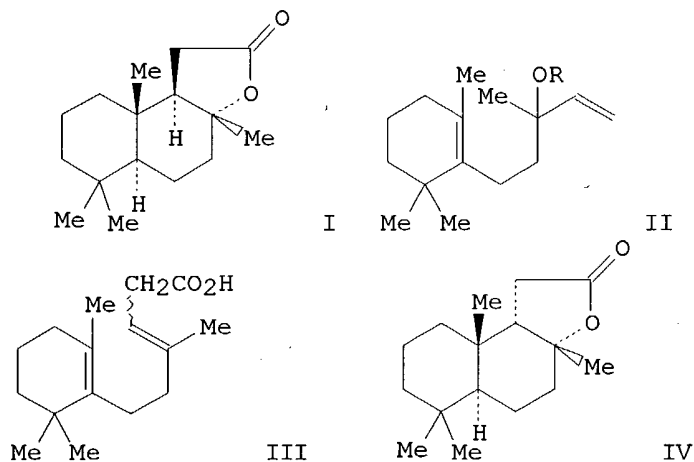
FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

Searcher : Shears 571-272-2528

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PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05186453	A2	19930727	JP 1991-358776	19911229
JP 3099317	B2	20001016		
PRIORITY APPLN. INFO.:			JP 1991-358776	19911229
OTHER SOURCE(S):			CASREACT 120:8779; MARPAT 120:8779	
GI				



AB (±)-Norambreinolide (I), a known tobacco smoke flavor enhancer and useful as an intermediate for ambrox, is prepared Thus, hydrogenation of β-ionone over Ni-diatomaceous earth at H pressure 10 atom and 80° in EtOH and addition of the resulting dihydro-β-ionone with CH<sub>2</sub>:CHMgCl in THF at 15-100° followed by hydrolysis with 5% aqueous H<sub>2</sub>SO<sub>4</sub> gave dihydro-β-vinyllionol II (R = H). Refluxing the latter with NaH in PhMe for 10 h followed by esterification with ClCO<sub>2</sub>Me and reaction of the resulting ester II (R = CO<sub>2</sub>Me) with CO 50 atm in the presence of 5% Pd-C and tri(o-tolyl)phosphine in isopropanol at 50-60° in an autoclave followed by saponification with 30% aqueous NaOH and acidification with 5% aqueous H<sub>2</sub>SO<sub>4</sub> gave a 33:67 mixture of cis- and trans-β-monocyclohomofarnesic acid (III) which was cyclized by the treatment with ClSO<sub>3</sub>H in CH<sub>2</sub>Cl<sub>2</sub> at -60 to -70° to give a 67:33 mixture of I and (±)-9-epi-norambreinolide (IV).

IT 151123-71-6P 151526-66-8P

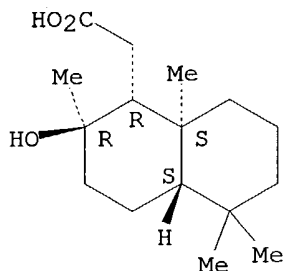
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of, from β-ionone)

RN 151123-71-6 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.

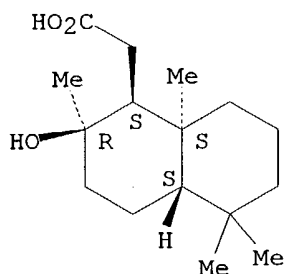
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RN 151526-66-8 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-,  
(1α,2α,4α,8αβ)- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L4 ANSWER 12 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1992:129310 CAPLUS

DOCUMENT NUMBER: 116:129310

TITLE: Synthesis of bicyclohomofarnesane derivatives of  
bis(8α,13-epoxy-14,15-bisnorlabd-12-en-12-  
yl)methane, a product of sclareol ozonolysis

AUTHOR(S): Aryku, A. N.; Koltsa, M. N.; Vlad, P. F.; Kukovinets,  
O. S.; Odionokov, V. N.; Tolstikov, G. A.

CORPORATE SOURCE: Inst. Khim., Kishinev, USSR

SOURCE: Khimiya Prirodnykh Soedinenii (1991), (3), 343-9  
CODEN: KPSUAR; ISSN: 0023-1150

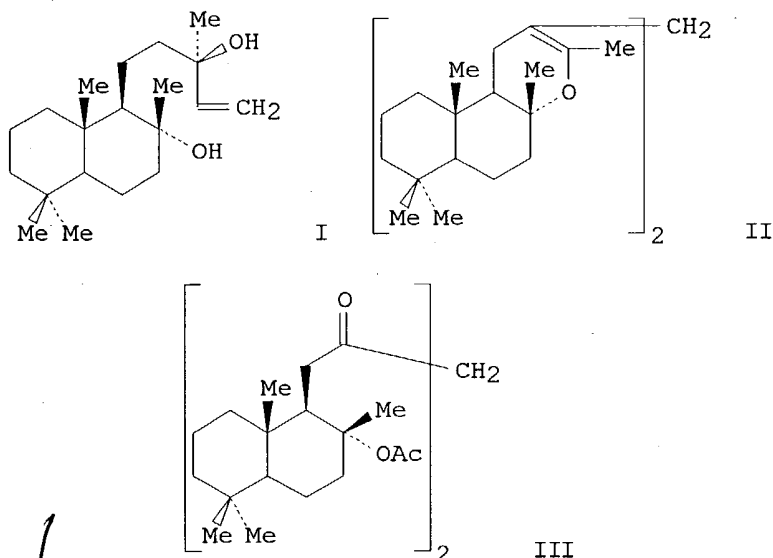
DOCUMENT TYPE: Journal

LANGUAGE: Russian

OTHER SOURCE(S): CASREACT 116:129310

GI

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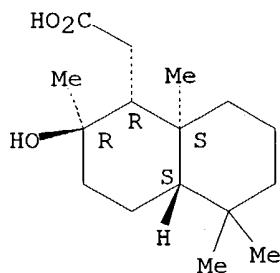
AB A method for obtaining novel fragrant analogs of ambroxide, with a strong  
amber odor, from Sclareol (I) is described. The key steps in the  
synthesis of these products are ozonolytic decomposition of I with the  
formation of bis(8 $\alpha$ ,13-epoxy-14,15-bisnorlabd-12-en-12-yl)methane  
(II), and its ozonization to bis(13,14,15,16-tetranorlabdan-8 $\alpha$ -  
acetoxy-12-on-12-yl)methane (III) followed by base-catalyzed decomposition  
IT **13456-36-5P**

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)  
(preparation and thermal cyclization of)

RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-,  
(1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L4 ANSWER 13 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1992:18368 CAPLUS  
DOCUMENT NUMBER: 116:18368

Searcher : Shears 571-272-2528

10/820709

TITLE: Diterpenes and norditerpenes from the Aristeguetia group  
AUTHOR(S): Zdero, C.; Bohlmann, F.; King, R. M.  
CORPORATE SOURCE: Inst. Org. Chem., Tech. Univ. Berlin, Berlin, D-1000/12, Germany  
SOURCE: Phytochemistry (1991), 30(9), 2991-3000  
CODEN: PYTCAS; ISSN: 0031-9422  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB A reinvestigation of Aristeguetia buddleaefolia gave 16 new labdanes and two nor-labdanes, while A. glutinosa afforded, in addition to large amts. of 8,15-dihydroxylabdane, three new norlabdanes. Badillao salicina gave six new cis-clerodanes and Grosvenoria rimbachii, guaianolides and tremetone derivs., four of which were new. The structures were elucidated by high field NMR techniques and chemical transformations. The chemotaxonomy is discussed briefly.

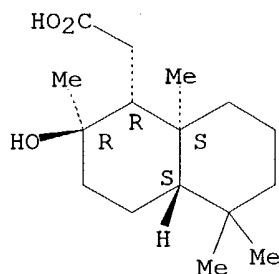
IT 13456-36-5

RL: BIOL (Biological study)  
(from Aristeguetia glutinosa)

RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 137960-53-3P

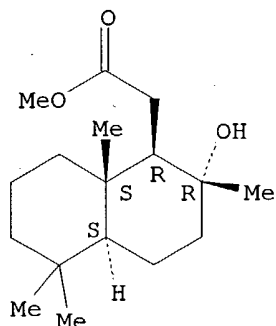
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 137960-53-3 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, methyl ester, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

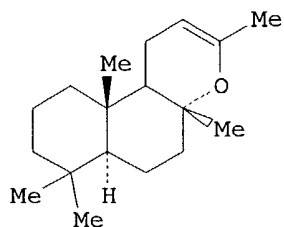
10/820709



L4 ANSWER 14 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 1991:536446 CAPLUS  
 DOCUMENT NUMBER: 115:136446  
 TITLE: Preparation of sclareolide  
 INVENTOR(S): Gerke, Thomas; Bruns, Klaus  
 PATENT ASSIGNEE(S): Henkel K.-G.a.A., Germany  
 SOURCE: Ger. Offen., 4 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3942358	A1	19910627	DE 1989-3942358	19891221
WO 9109852	A1	19910711	WO 1990-EP2166	19901213
W: CA, JP, US				
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LU, NL, SE				
EP 506776	A1	19921007	EP 1991-901525	19901213
EP 506776	B1	19940914		
R: CH, DE, ES, FR, GB, LI, NL				
JP 05502232	T2	19930422	JP 1991-501820	19901213
JP 3020272	B2	20000315		
ES 2060354	T3	19941116	ES 1991-901525	19901213
US 5247100	A	19930921	US 1992-862560	19920622
PRIORITY APPLN. INFO.:			DE 1989-3942358	A 19891221
			WO 1990-EP2166	W 19901213

GI



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Searcher : Shears 571-272-2528

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AB Sclareol was converted to sclareolide in 65% overall yield by oxidative degradation with NaOCl-RuO under phase-transfer conditions, H<sub>2</sub>O<sub>2</sub> oxidation of the resulting enol ether I and lactonization of the resulting hydroxy acid by heating.

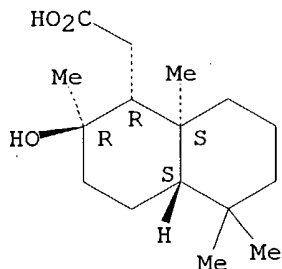
IT 13456-36-5P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation and lactonization of)

RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L4 ANSWER 15 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1985:557322 CAPLUS

DOCUMENT NUMBER: 103:157322

TITLE: Structure of new bromoditerpenes, pinnatols, from the marine red alga Laurencia pinnata Yamada  
AUTHOR(S): Fukuzawa, Akio; Miyamoto, Mitsuaki; Kumagai, Yoshikazu; Abiko, Atsushi; Takaya, Yoshiaki; Masamune, Tadashi

CORPORATE SOURCE: Fac. Sci., Hokkaido Univ., Sapporo, 060, Japan

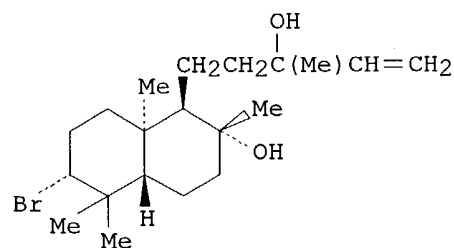
SOURCE: Chemistry Letters (1985), (8), 1259-62

CODEN: CMLTAG; ISSN: 0366-7022

DOCUMENT TYPE: Journal

LANGUAGE: English

GI



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AB The structure of new bromoditerpenes, named pinnatol A (I), B, C, and D, isolated from *L. pinnata*, was determined on the basis of the chemical and spectral data.

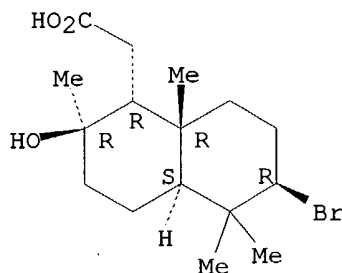
IT 98687-64-0P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation and reaction of)

RN 98687-64-0 CAPLUS

CN 1-Naphthaleneacetic acid, 6-bromodecahydro-2-hydroxy-2,5,5,8a-tetramethyl-, [1R-(1 $\alpha$ ,2 $\beta$ ,4 $\alpha\alpha$ ,6 $\beta$ ,8 $\alpha\beta$ )]- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L4 ANSWER 16 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1981:15913 CAPLUS

DOCUMENT NUMBER: 94:15913

TITLE: Improved method for the reduction of norambreinolide

AUTHOR(S): Sibirtseva, V. E.; Kustova, S. D.; Tokareva, V. Ya.; Vlad, P. F.; Koltsa, M. N.

CORPORATE SOURCE: Vses. Nauchno-Issled. Inst. Sint. Nat. Dushistyykh Veshchestv, Moscow, USSR

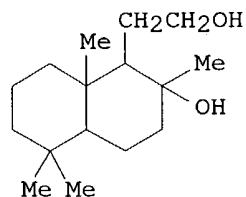
SOURCE: Maslozhirovaya Promyshlennost (1980), (7), 29-30

CODEN: MZPYAE; ISSN: 0025-4649

DOCUMENT TYPE: Journal

LANGUAGE: Russian

GI



I

AB Reduction of norambreinolide by a mixture containing KBH4 and LiCl gave 64-5% the

Searcher : Shears 571-272-2528



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diol I of 82-5% purity.

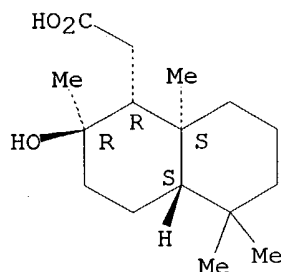
IT **13456-36-5P**

RL: FORM (Formation, nonpreparative); PREP (Preparation)  
(formation of, in reduction of norambreinolide)

RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-,  
(1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L4 ANSWER 17 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1971:125869 CAPLUS

DOCUMENT NUMBER: 74:125869

TITLE: Diterpenoids. XXVIII. Synthesis of  
 $\alpha$ -onoceradiene from abienol

AUTHOR(S): Carman, Raymond M.; Deeth, H. C.

CORPORATE SOURCE: Chem. Dep., Univ. Queensland, St. Lucia, Australia

SOURCE: Australian Journal of Chemistry (1971), 24(5),  
1099-102

CODEN: AJCHAS; ISSN: 0004-9425

DOCUMENT TYPE: Journal

LANGUAGE: English

GI For diagram(s), see printed CA Issue.

AB The structure of  $\alpha$ -onoceradiene (I) is confirmed by the preparation of I from abienol (II) in a series of reactions. Thus, II is treated with KMnO<sub>4</sub> to give norambreinolide which is hydrolyzed to III. IV is dehydrated to Me 13,14,15,16-tetranorlabd-8(17)-en-12-oate (V). I [[ $\alpha$ ]D 28° (hexane)] is prepared by the electrolysis of VI in MeOH containing NaOMe.

IT **13456-36-5P**

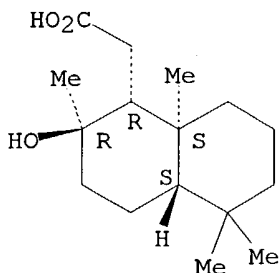
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-,  
(1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

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L4 ANSWER 18 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1966:508201 CAPLUS

DOCUMENT NUMBER: 65:108201

ORIGINAL REFERENCE NO.: 65:20170a-c

TITLE: Optical rotation and structure in the labdane series of diterpenoids

AUTHOR(S): Carman, R. M.

CORPORATE SOURCE: Univ. Queensland, Brisbane

SOURCE: Australian Journal of Chemistry (1966), 19(4), 629-42  
CODEN: AJCHAS; ISSN: 0004-9425

DOCUMENT TYPE: Journal

LANGUAGE: English

GI For diagram(s), see printed CA Issue.

AB Correction of CA 65, 7219g. On the basis of mol. rotations of 143 labdane (I) derivs. (tabulated) the correlation between their optical rotation and their structure was discussed. Sclareol (II) was hydrogenated in EtOH over Pd-C at 3 atmospheric H pressure to give dihydrosclareol, m. 114-15°,  $[\alpha]_D -1.3^\circ$  (c 0.9, all in CHCl<sub>3</sub>). Biformene was hydrogenated in C<sub>6</sub>H<sub>14</sub> over Adams' catalyst at atmospheric pressure and 25° to give a product with  $[\alpha]_D 30^\circ$  (c 0.8), which on further hydrogenation in EtOH over Pd-C at 2 atmospheric gave hexahydrobiformene (8S, 13 $\xi$ -labdane),

b0.25 96°, n<sub>20D</sub> 1.4920, d<sub>20</sub> 0.910,  $[\alpha]_D 36^\circ$  (c 1.3).

Dehydration of II with p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H (Ruzika and Janot, CA 25, 3658) gave an oil, b0.07 108°,  $[\alpha]_D -6.0^\circ$  (c 0.3), n<sub>17D</sub> 1.5224, d<sub>17</sub> 0.940, which could not be purified by repeated chromatography. The results indicated that there is a simple relation between rotation and structure in the labdane series of terpenoids.

IT 10314-52-0, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-

2,5,5,8 $\alpha\beta$ -tetramethyl-, (2S)- 10314-53-1,

1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8 $\alpha\beta$ -tetramethyl-,

methyl ester, (2S)- 13456-36-5, 1-Naphthaleneacetic acid,

decahydro-2-hydroxy-2,5,5,8 $\alpha\beta$ -tetramethyl-, (2R)-

(optical rotation of)

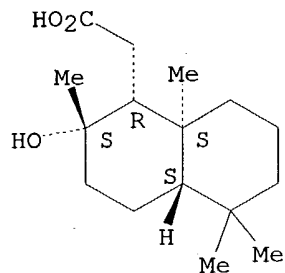
RN 10314-52-0 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8 $\alpha\beta$ -tetramethyl-,

(2S)- (8CI) (CA INDEX NAME)

Absolute stereochemistry.

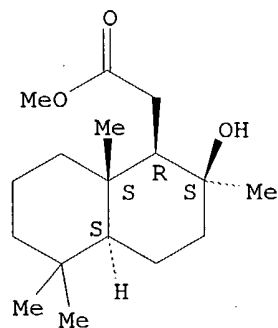
10/820709



RN 10314-53-1 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, methyl ester, [1R-(1α,2α,4aβ,8aα)]- (9CI) (CA INDEX NAME)

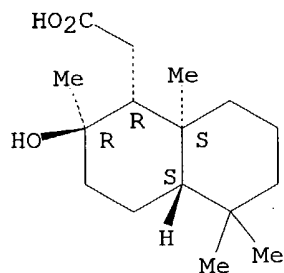
Absolute stereochemistry.



RN 13456-36-5 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, (1R,2R,4aS,8aS)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L4 ANSWER 19 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1964:2846 CAPLUS  
DOCUMENT NUMBER: 60:2846

Searcher : Shears 571-272-2528

10/820709

ORIGINAL REFERENCE NO.: 60:431c-f  
 TITLE: Resolution of racemates of bicycloalicyclic compounds  
 INVENTOR(S): Prelog, Vladimir  
 PATENT ASSIGNEE(S): CIBA Ltd.  
 SOURCE: 5 pp.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CH 366530		19630228	CH	19560327

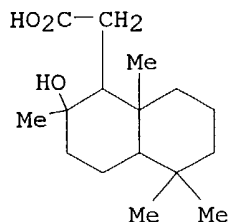
AB Racemates of bicycloalicyclic compds., particularly hydronaphthalene and hydroindene derivs., containing at least 1 carbonyl group, are resolved by reducing them with suitable microorganisms and isolating the diastereoisomeric hydroxy compds., which are oxidized to the antipodes of the original oxo compound. Thus, a solution of 40 g. cane sugar, 40 g. Difco Trypton, 8 g. NaNO<sub>3</sub>, 4 g. K<sub>2</sub>HPO<sub>4</sub>, 2 g. MgSO<sub>4</sub>, 2 g. KCl, and 40 mg. FeSO<sub>4</sub> in 4 l. H<sub>2</sub>O was brought to pH 7, treated with 10 g. CaCO<sub>3</sub>, sterilized, and inoculated with a culture of Curvularia falcata. After 3 days at 27°, 1 g. dl-Δ<sub>4</sub>,10-3,8-dioxo-9-methyloctalin (dl-I) in 15 cc. acetone was added, the mixture shaken 3 days, filtered, the filtrate extracted with AcOEt, the extract washed with dilute HCl, KHCO<sub>3</sub>, and H<sub>2</sub>O, and evaporated to dryness in vacuo. The residue was chromatographed on Al<sub>2</sub>O<sub>3</sub> with benzene. The first fractions were optically inactive and contained I, the middle fractions contained optically active compound, C<sub>18</sub>H<sub>21</sub>O<sub>6</sub>N (p-nitrobenzoate m. 106.5-7.5°, [α]<sub>D</sub> -26°), and the last fractions contained (+)-Δ<sub>4</sub>,10-3-oxo-8-hydroxy-9-methyl-octalin [(+)-II], [α]<sub>D</sub> 203° (p-nitrobenzoate m. 195°, [α]<sub>D</sub> 159°). The column was then eluted with Et<sub>2</sub>O-AcOEt mixts. to give (-)-II, m. 94-5°, [α]<sub>D</sub> -129° (p-nitrobenzoate m. 122.5°, [α]<sub>D</sub> 87°). A solution of 250 mg. (-)-II in 4 cc. pyridine was treated with 340 mg. CrO<sub>3</sub>, kept 2 days at room temperature, diluted with H<sub>2</sub>O, the solution extracted with C<sub>6</sub>H<sub>6</sub>-Et<sub>2</sub>O, the extract evaporated, and the residue adsorbed on Al<sub>2</sub>O<sub>3</sub> with C<sub>6</sub>H<sub>6</sub>-petr. ether to give (-)-I, m. 50.5°, [α]<sub>D</sub> -100°. Similarly was prepared (+)-I, m. 50°, [α]<sub>D</sub> 100°. The same method was used to reduce 430 mg. dl-Δ<sub>4</sub>,9-1,5-dioxo-8-methylhexahydroindene (dl-III), but the benzene eluate contained l-III, m. 58-60°, [α]<sub>D</sub> -312° (benzene). The residue was eluted with 19:1 C<sub>6</sub>H<sub>6</sub>-Et<sub>2</sub>O to give mainly d-Δ<sub>4</sub>,9-5-oxo-1-hydroxy-8-methylhexahydroindene (IV), and with 4:1 C<sub>6</sub>H<sub>6</sub>-Et<sub>2</sub>O, some l-IV. Other examples used Ophiobolus herpotrichus, Rhizopus nigricans, and Streptomyces coelicolor.

IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (preparation of)

RN 93158-29-3 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI, 7CI) (CA INDEX NAME)

10/820709



L4 ANSWER 20 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1963:8782 CAPLUS  
DOCUMENT NUMBER: 58:8782  
ORIGINAL REFERENCE NO.: 58:1435b-d  
TITLE: Two-stage oxidation of sclareol  
INVENTOR(S): Schumacher, Joseph N.; Henley, Walter M.; Bernasek,  
Edward; Teague, Claude E. Jr.  
PATENT ASSIGNEE(S): R. J. Reynolds Tobacco Co.  
SOURCE: 4 pp.  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3050532		19620821	US	19590619

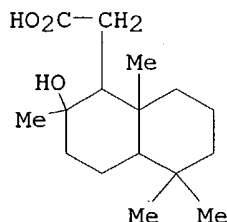
AB To sclareol (205.6 g.) and 1435 cc. H<sub>2</sub>O was added 379.5 g. KMnO<sub>4</sub> in 3 hrs., the mixture further agitated at 30-5° 2 hrs., to this 1435 cc. AcOH added with further agitation, 254 g. KMnO<sub>4</sub> in 104 cc. AcOH added in 2 hrs. at 8-10°, the mixture kept 15 hrs. at room temperature, 1 l. H<sub>2</sub>O added, the mixture acidified to pH 2 with H<sub>2</sub>SO<sub>4</sub>, cooled to 10°, SO<sub>2</sub> passed in to convert the precipitated MnO<sub>2</sub> to the H<sub>2</sub>O-soluble MnSO<sub>4</sub>, the organic mixture separated from the aqueous phase, the dried product hydrolyzed with 110 g. KOH in 150 cc. H<sub>2</sub>O and 1500 cc. MeOH 3 hrs., the MeOH removed, the residue from the distillation dissolved in 2 l. H<sub>2</sub>O, the solution washed with C<sub>6</sub>H<sub>12</sub> and the aqueous layer acidified with 6N H<sub>2</sub>SO<sub>4</sub> to pH 2 to give 2-hydroxy-2,5,5,8a-tetramethyldecahydro-1-naphthaleneacetic acid (I). I was heated 1 1/2-2 hrs. at 135-45° to give 65% decahydro-3a,6,6,9a-tetramethylnaphtho[2,1-b]furan-2(1H)-one (II), m. 123-4° (C<sub>6</sub>H<sub>12</sub>).

IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-  
(preparation of)

RN 93158-29-3 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI, 7CI) (CA INDEX NAME)

10/820709



L4 ANSWER 21 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 1960:13261 CAPLUS  
 DOCUMENT NUMBER: 54:13261  
 ORIGINAL REFERENCE NO.: 54:2679a-c  
 TITLE: Improving the flavor of tobacco  
 INVENTOR(S): Schumacher, Joseph N.  
 PATENT ASSIGNEE(S): R. J. Reynolds Tobacco Co.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2905576		19590922	US	
DE 1209473			DE	
GB 847201			GB	
GB 847201			GB	

AB The addition of 0.01-1.0%, preferably 0.05-0.3%, of a compound selected from the group of 2-hydroxy-2,5,5,8a-tetramethyldecahydro-1-naphthaleneacetic acid (III), the lactone of III (IV), and decahydro-3a,6,6,9a-tetramethylnaphtho[2,1-b]furan-2-one (V) to domestic tobacco imparts also a cedarlike odor. Thus, V was prepared from a solution of IV 5 in CCl4 30

by adding N-bromosuccinimide 3.6 parts, refluxing for 30 min., filtering, and evaporation of the filtrate. The brominated derivative of IV, (m. 129-31°), was recovered by chromatography on silicic acid with elution by 3:1 C6H6-petr. ether. A solution of 5 parts of the brominated derivative of IV

in 45 parts 2,4,6-trimethylpyridine was refluxed for 1 hr., cooled, and diluted with Et2O. The Et2O solution was washed with dilute HCl and H2O and evaporated V,

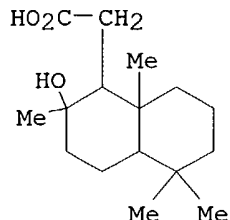
(m. 122.5°), was recovered from the concentrate by chromatography on SiO2 gel and elution with C6H6. The additive can be applied to tobacco in a solution or suspension by spraying, dipping etc.

IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-  
 (as flavoring material for tobacco)

RN 93158-29-3 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI, 7CI) (CA INDEX NAME)

10/820709



L4 ANSWER 22 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1959:100021 CAPLUS

DOCUMENT NUMBER: 53:100021

ORIGINAL REFERENCE NO.: 53:18093b-f

TITLE: C $\beta$ -C $\gamma$  Cleavage of a  $\gamma$ -hydroxy acid by electrolytic oxidation

AUTHOR(S): Corey, E. J.; Sauers, R. R.

CORPORATE SOURCE: Univ. of Illinois, Urbana

SOURCE: Journal of the American Chemical Society (1959), 81, 1743-5

CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 53:100021

GI For diagram(s), see printed CA Issue.

AB cf. preceding abstract A novel elimination process, which has been observed during anodic reaction of a  $\gamma$ -HO acid, is described. The vinyl ketone obtained by the electrolysis in MeOH of I (R = Me, R' = OH) (as NH<sub>4</sub> salt) (cf. preceding abstract) was identified as 4-(1,3,3-trimethyl-1-vinyl-2-cyclohexyl)-2-butanone (II), b<sub>0.4</sub> 95-6°, n<sub>21D</sub> 1.4857, [ $\alpha$ ]<sub>26D</sub> -10.4° (c 1.06); semicarbazone, m. 175.5-8.5°, plates from aqueous EtOH. II, also obtained in the electrolysis of the NH<sub>4</sub> salt of I (R = OH, R' = Me), b<sub>0.3</sub> 93-100°, n<sub>24.5D</sub> 1.4834. Br (0.8 cc.) added to 1.8 g. NaOH in 14 cc. H<sub>2</sub>O, a 2.9-cc. portion added to 0.204 g. II in 14 cc. H<sub>2</sub>O, stirred 12 hrs. at room temperature, heated 15 min. on

the steam bath, poured into 25 cc. H<sub>2</sub>O, extracted with Et<sub>2</sub>O, and the aqueous phase

acidified with dilute HCl and extracted with Et<sub>2</sub>O gave 0.150 g. 3-(1,3,3-trimethyl-1-vinyl-2-cyclohexyl)propionic acid; benzylisothiuronium salt, plates, m. 143-5° (aqueous EtOH). II (0.432 g.) in 10 cc. MeOH hydrogenated over 39 mg. 5% Pd-C, evaporated in vacuo, diluted with H<sub>2</sub>O, and extracted with Et<sub>2</sub>O, the extract worked up, and the residual

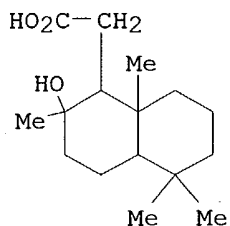
oil chromatographed on Al<sub>2</sub>O<sub>3</sub> gave 0.286 g. dihydro-II, clear oil; semicarbazone, m. 156.5-8.5°.

IT 109727-79-9, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, ammonium salt (preparation of)

RN 109727-79-9 CAPLUS

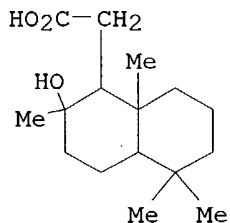
CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-, ammonium salt (6CI) (CA INDEX NAME)

10/820709



●-NH<sub>3</sub>

IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (stereoisomers)  
RN 93158-29-3 CAPLUS  
CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI, 7CI) (CA INDEX NAME)



L4 ANSWER 23 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1958:25433 CAPLUS  
DOCUMENT NUMBER: 52:25433  
ORIGINAL REFERENCE NO.: 52:4580i,4581a-b  
TITLE: Carbon-β-carbon-γ cleavage of a γ-hydroxy acid by electrolytic oxidation  
AUTHOR(S): Corey, E. J.; Sauers, Ronald R.; Swann, Sherlock, Jr.  
CORPORATE SOURCE: Univ. of Illinois, Urbana  
SOURCE: Journal of the American Chemical Society (1957), 79, 5826-7  
CODEN: JACSAT; ISSN: 0002-7863  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable  
GI For diagram(s), see printed CA Issue.  
AB cf. C.A. 52, 1110h. An elimination process is described which occurs concurrently with the conversion of salts of I (R<sub>1</sub> = Me, R<sub>2</sub> = OH) and I (R<sub>1</sub> = OH, R<sub>2</sub> = Me) (II) to tetracyclic triterpenes (loc. cit.) and leads to 34-8% IV, b<sub>0.4</sub> 95-6°, n<sub>D</sub> 1.4857, [α]<sub>D</sub> 26D -10.4 (CHCl<sub>3</sub>) (semicarbazone, m. 175.5-8.5°); hydrogenation over Pd-C in MeOH gave a dihydroketone; semicarbazone, m. 156.5-8.5°. IV with NaOBr yielded a liquid nor acid; benzylthiuronium salt, m. 143-5°. Electrolytic reduction of I (R<sub>1</sub> = Me, R<sub>2</sub> = OAc) (III) yielded the onocerane coupling product but no IV. Electrolytic oxidation of II also yielded IV

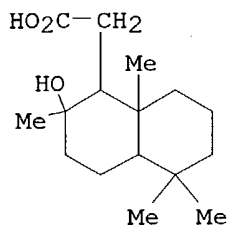
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plus considerable amts. of 2 similar ketonic substances, possibly formed from V by migration of H and Me and subsequent elimination. IV prepared from I was usually contaminated with about 3% of a closely similar ketonic impurity (probably isomeric).

IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-  
(stereoisomers, electrolytic oxidation-reduction of)  
RN 93158-29-3 CAPLUS  
CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI, 7CI) (CA INDEX NAME)



L4 ANSWER 24 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1955:60419 CAPLUS

DOCUMENT NUMBER: 49:60419

ORIGINAL REFERENCE NO.: 49:11609b-d

TITLE: Odor and constitution. XII. Influence of the steric configuration on the semireduction of lactones by lithium aluminum hydride

AUTHOR(S): Hinder, M.; Stoll, M.

CORPORATE SOURCE: Firmenich & Cie., Geneva, Switz.

SOURCE: Helvetica Chimica Acta (1954), 37, 1866-71

CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

LANGUAGE: French

AB The isolactone (I), m. 93°, obtained by isomerization of the lactone (II) of 1,1,6,10-tetramethyl-6-hydroxy-5-decalylacetic acid gives with LiAlH<sub>4</sub> at -30° an isohemiacetal (III), m. 92.5-3.5°, purified by chromatography on Al<sub>2</sub>O<sub>3</sub>. Heating III 65 hrs. at 67-90° in vacuo or distilling slowly a C<sub>6</sub>H<sub>6</sub> solution of III and 2-C<sub>10</sub>H<sub>7</sub>SO<sub>3</sub>H (IV) gives

an isoanhydride (V), m. 170-70.5°. V refluxed with IV in MeOH gives the Me ether of III (VI), m. 52-2.5°, [ $\alpha$ ]<sub>D</sub><sup>25</sup> -72.9 ± 1.5° (c 10.42, C<sub>6</sub>H<sub>6</sub>). VI and 2,4-(NO<sub>2</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NHNH<sub>2</sub> give a 2,4-dinitrophenylhydrazone, m. 121-3.5°. VI heated with MeOH-H<sub>2</sub>SO<sub>4</sub> gives III directly. Infrared spectra are reported for these compds. II and LiAlH<sub>4</sub> give a glycol, m. 131-2°. A similar reaction with pentadecanolide also gives a glycol, m. 88-8.5°. Thus the reaction of semireduction of lactones is not general. It is related to the steric configuration of  $\gamma$ -lactones.

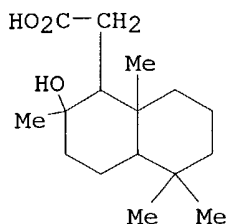
IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-

(stereoisomers,  $\gamma$ -lactones, reduction of)

RN 93158-29-3 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI,

7CI) (CA INDEX NAME)



L4 ANSWER 25 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1955:60418 CAPLUS

DOCUMENT NUMBER: 49:60418

ORIGINAL REFERENCE NO.: 49:11609a-b

TITLE: Odor and constitution. XI. The transesterification-dehydration of the lactone of 1,1,6,10-tetramethyl-6-hydroxy-5-decalylacetic acid

AUTHOR(S): Stoll, M.; Hinder, M.

CORPORATE SOURCE: Firmenich &amp; Cie., Geneva, Switz.

SOURCE: Helvetica Chimica Acta (1954), 37, 1859-66

CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

LANGUAGE: French

AB The lactone (I) refluxed 21 hrs. with MeOH-H<sub>2</sub>SO<sub>4</sub> gives the free acid (II), m. 118-18.6°, [α]<sub>D</sub><sup>26</sup> -23.7 ± 1.5° (c 3.2, C<sub>6</sub>H<sub>6</sub>); p-phenylphenacyl ester, m. 91.2-1.7°; Me ester, b0.04 107-8°, m. about 30°. II hydrogenated over Pt gives a saturated acid, m. 107-7.5°. When I is refluxed 24 hrs. with MeOH-H<sub>2</sub>SO<sub>4</sub> it is partly isomerized to an isolactone (III), m. 92-3°, and an acid (IV) isomeric with II. II and IV form a eutectic, m. 84°, which cannot be separated by chromatography on Al<sub>2</sub>O<sub>3</sub>. Refluxing 96 hrs. gives

pure IV, m. 102°; p-phenylphenacyl ester, m. 103-4°. IV cannot be hydrogenated at ordinary pressures. Hydrolysis of III produces an ester, b0.01 98-9°. I hydrolyzes more easily than III and the mixed esters of II and IV are saponified still more slowly. Infrared spectra are reported for the various compds.

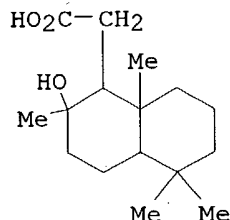
IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-

(stereoisomers, and their γ-lactones, transesterification and dehydration)

RN 93158-29-3 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI, 7CI) (CA INDEX NAME)

10/820709



L4 ANSWER 26 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1955:4838 CAPLUS

DOCUMENT NUMBER: 49:4838

ORIGINAL REFERENCE NO.: 49:1025c-h

TITLE: Odor and constitution. IX. Preparation of bicyclohomofarnesic stereoisomeric substances

AUTHOR(S): Hinder, M.; Stoll, M.

CORPORATE SOURCE: Maison Firmenich & Cie., Geneva, Switz.

SOURCE: Helvetica Chimica Acta (1953), 36, 1995-2008

CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

LANGUAGE: French

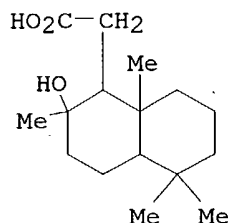
GI For diagram(s), see printed CA Issue.

AB Isosclareol (I) should exist in the mother liquors from sclareol (II), since in the degradation product of II which gives a normal glycol (III), m. 132°, there is a little isoglycol (IV), m. 191°. No I is found by chromatography of crude II. Oxidation of crude II to the lactone and its reduction give only III. Thus I does not occur naturally with II. Compds. of the iso series are formed when the normal lactone. (V), m. 125°, is treated with H2SO4 to give some isolactone (VI), m. 92.5-3.5°, reduced by LiAlH4 to IV. Isomerization also occurs at high temperature in the absence of H2SO4. KOH sapons. V to a hydroxy acid, m.

128° (Me ester, m. 80-2.5°), and VI to the isohydroxy acid (VII), m. 128° (Me ester, m. 114-15°). This saponification is 4 times slower. LiAlH4 reduces VII to IV which, heated with 2-ClOH7SO3H gives a mixture of the isoeopoxide (VIII), m. 59-60° and the normal epoxide (IX), m. 75°. IX can be partly isomerized to VIII with HOAc-H2SO4 in a sealed tube; 9-12% of a hydrocarbon b0.003 75-87° is also formed. II and III do not isomerize under these conditions, but are dehydrated. Attempts to isomerize sclareol oxide give a mixture of hydrocarbons C18H28, b0.003 101-3°, d20.4 0.9736, nD20 1.5407, MRD calculated 77.79, found 78.82, absorption maximum 240,232 mμ, log ε 4.29, 4.25. About 20% of this mixture resists hydrogenation and is probably X. The mixture also contains compds. m. 92-3° and 122-6° of unknown composition and a ketone (XI) b0.005 105°, d19.4 0.9736, nD20 1.5088, MRD calculated 80.46, found 80.48 (2,4-dinitrophenylhydrazone m. 151-2.5°), probably identical with the compound prepared by Ruzicka, Seidel, and Engel (C.A. 37, 877.3). Attempts to convert XI to the epoxide and to reduce this to the isoglycol give a mixture from which no tryst, compound is obtained. KMnO4 oxidation of II gives XII, m. 87-8.5°, which with LiAlH4 gives a mixture of stereoisomeric glycols m. 102-2.6° and 112-13°. IV with CrO3 gives only VI. Infrared spectra are reported for the various compds. described.

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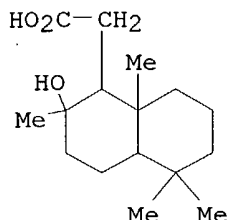
IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-  
(stereoisomers, and their  $\gamma$ -lactones and other derivs.)  
RN 93158-29-3 CAPLUS  
CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI, 7CI) (CA INDEX NAME)



L4 ANSWER 27 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1955:4837 CAPLUS  
DOCUMENT NUMBER: 49:4837  
ORIGINAL REFERENCE NO.: 49:1024g-i,1025a-c  
TITLE: Odor and constitution. VIII. Some products of the degradation of sclareol  
AUTHOR(S): Stoll, M.; Hinder, M.  
CORPORATE SOURCE: Maisin Firmenich & Cie., Geneva, Switz.  
SOURCE: Helvetica Chimica Acta (1953), 36, 1984-95  
CODEN: HCACAV; ISSN: 0018-019X  
DOCUMENT TYPE: Journal  
LANGUAGE: French  
GI For diagram(s), see printed CA Issue.  
AB cf. C.A. 47, 3526c. Crude sclareol (I) contains some n-nonacosane, m. 63-5°. The ozonide of I oxide refluxed with H<sub>2</sub>O gives chiefly an acetyl hydroxy aldehyde, C<sub>18</sub>H<sub>30</sub>O<sub>3</sub> (II). Distillation of the residual neutral fraction which contains mostly the unsatd. aldehyde (C.A. 45, 1551h) causes its decomposition to a lactone (III), m. 215-15.5°. LiAlH<sub>4</sub> converts III to a glycol (IV), m. 131.5-2.5°, and hydrolysis of III gives a mixture of AcH, the normal lactone (V), m. 123-4°, and the HO acid (VI), m. 126-7°. VI when treated with AcH gives only V. II and reagent D of Viscontini and Meier (C.A. 45, 3802e) in glacial AeOH give a mixture of the N,N-dimethylglycine hydrazone (VII) of II, m. 138-9° and a compound C<sub>26</sub>H<sub>48</sub>O<sub>3</sub>N<sub>6</sub>, m. 219.5-20.2° (decomposition). VII in Et<sub>2</sub>O with H<sub>2</sub>SO<sub>4</sub> gives II (semicarbazone, m. 207°). II with KMnO<sub>4</sub> gives the AcO acid, m. 156.5-7.5° (Me ester m. 71-2°). KOH saponification of II gives a hemiacetal (VIII), m. 118-19°. VIII and KMnO<sub>4</sub> give V. VIII sublimes slowly in a high vacuum leaves a residue which has formed an anhydride, m. 216° (decomposition), which regenerates VIII when heated with 20% H<sub>2</sub>SO<sub>4</sub> in dioxane. IV with CrO<sub>3</sub> in tert-BuOH gives no aldehyde, but 82% V. Attempts at partial reduction of V with LiAlH<sub>4</sub> give only IV. IR spectra are reported for all these compds.  
IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-  
(stereoisomers, and their  $\gamma$ -lactones and other derivs.)  
RN 93158-29-3 CAPLUS

10/820709

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI, 7CI) (CA INDEX NAME)



L4 ANSWER 28 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1951:8682 CAPLUS

DOCUMENT NUMBER: 45:8682

ORIGINAL REFERENCE NO.: 45:1551h-i,1552a-b

TITLE: Odor and constitution. III. Bicyclohomofarnesic substances

AUTHOR(S): Stoll, M.; Hinder, M.

CORPORATE SOURCE: Firmenich et Cie, Geneva, Switz.

SOURCE: Helvetica Chimica Acta (1950), 33, 1251-60

CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

LANGUAGE: French

AB cf. C.A. 44, 1069h. Sclareol oxide with O<sub>3</sub> gives an ozonide which with Raney Ni in EtOAc gives some acetylated HO acid, m. 166-7° [free HO acid (I), m. 126-8° identical with that obtained by Ruzicka, et al., C.A. 37, 877.3], neutral crystals, m. 224-6°, of undetd. composition, and a mixture which cannot be completely separated by fractionation. This contains a glycol monoacetate [free glycol (II), m. 130-2°] and an acetylated HO aldehyde (III) which during saponification in air is oxidized to I (lactone, m. 123-4°). If the saponification is carried out in N, III gives a free HO aldehyde (IV) (partly purified semicarbazone, m. 134-6°). Some anhydride of IV is also formed. Complete purification of these compds. is not possible. IV with Ag<sub>2</sub>O gives I. Distillation of III over Cu bronze gives

70% bicyclohomofarnesal (V), b<sub>0</sub>.008 100-3°, d<sub>19</sub>.24 0.9938, n<sub>19</sub>.5D 1.5130, MRD calculated 71.23, found 70.89 (semicarbazone, m. 223-5°), which is reduced over Pt to a saturated alc. (VI), b<sub>0</sub>.005 105° (3,5-dinitrobenzoate, m. 118-19°), with a weak odor. Reduction of III gives II which, heated with 2-ClOH<sub>7</sub>SO<sub>3</sub>H gives 1,1,-4a,6-tetramethyl-5-ethyl-6,52-oxidodecahydronaphthalene, m. 75-6°. The Ac derivative of I with Cu bronze gives an unsatd. acid which is reduced by LiAlH<sub>4</sub> to bicyclohomofarnesol (VII), b<sub>0</sub>.01 116-17° (dinitrobenzoate, m. 133-4.5°). V and VII are the compds. which give the "odor of sclareol" to these mixts.

IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (and derivs.)

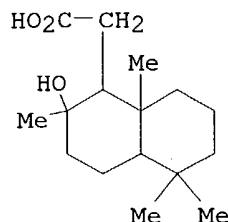
RN 93158-29-3 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI,

Searcher : Shears 571-272-2528

10/820709

7CI) (CA INDEX NAME)



L4 ANSWER 29 OF 29 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1943:5042 CAPLUS

DOCUMENT NUMBER: 37:5042

ORIGINAL REFERENCE NO.: 37:877b-i

TITLE: Diterpenes. LIII. Oxidation of sclareol with potassium permanganate

AUTHOR(S): Ruzicka, L.; Seidel, C. F.; Engel, L. L.

SOURCE: Helvetica Chimica Acta (1942), 25, 621-30

CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 37:5042

AB cf. C. A. 32, 4992.3. A structural formula has been proposed for sclareol (I), C<sub>20</sub>H<sub>36</sub>O<sub>2</sub>, the diterpene alc. from the leaves of *Salvia sclarea*, L. and new results confirming this structure are reported. 1 (60 g.) in 400 cc. acetone was treated at 0° with 105 g. KMnO<sub>4</sub> in 4 l. acetone and the colorless reaction product was filtered free from MnO<sub>2</sub> and washed with dilute NaOH. The alkaline filtrate was freed from acetone and the residue

was

extracted with ether. Acidification of the alkaline solution and shaking out with

ether gave 12 g. of the previously described di-HO acid (II), C<sub>19</sub>H<sub>34</sub>O<sub>4</sub>, m. 153-4°. The ether-soluble neutral oxidation product was digested with low-boiling petr. ether. Recrystn. of the petr. ether-insol. portion (10.3 g.) gave the HO ketone (III), C<sub>18</sub>H<sub>32</sub>O<sub>2</sub>, m. about 80°; semicarbazone, C<sub>19</sub>H<sub>35</sub>N<sub>3</sub>O<sub>2</sub>, m. 144-5°. High-vacuum distillation of the petr. ether-soluble neutral oxidation product and recrystn. from MeOH gave

15

g. of a mixture, m. 45-6°, yielding, on distillation, the unsatd. oxide (IV), C<sub>18</sub>H<sub>30</sub>, b<sub>10</sub> 174-6°, converted by treatment with H<sub>2</sub>NNHCONH<sub>2</sub>.AcOH into the semicarbazone of III and by boiling with dil alc. into III by cleavage of the oxide ring. Catalytic hydrogenation of IV in the presence of PtO<sub>2</sub> with 1 mol. H gave a mixture of stereoisomeric dihydro oxides, C<sub>18</sub>H<sub>32</sub>O, m. about 84°, which gave no semicarbazone. As expected, the dehydrogenation of IV by refluxing with Se for 33 hrs. at 340-50° gave 1,5,6-trimethylnaphthalene. Ozonization of 32 g. IV in 5-6 g. portions in 8-fold amts. of purified hexane, working up and recrystn. from 80% MeOH yielded 9 g. of acetoxy acid (V), C<sub>18</sub>H<sub>30</sub>O<sub>4</sub>, m. 157-8°. Saponification of V with alc. KOH, acidification, extraction with

ether

and recrystn. from petr. ether gave a crystalline HO acid (VI), m. 128-9°. By heating for 1 hr. at 130-50° under vacuum, VI

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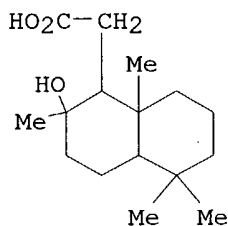
was lactonized. The crude residue was taken up in ether, washed with ice-cold Na<sub>2</sub>CO<sub>3</sub> and evaporated down. Recrystn. from petr. ether gave 5.6 g. of lactone (VII), C<sub>16</sub>H<sub>26</sub>O<sub>2</sub>, m. 123-4°, [α]<sub>D</sub> 45.9° (c 3.0 in CHCl<sub>3</sub>), giving no m.-p. lowering with the lactone obtained by the oxidation of I with CrO<sub>3</sub>. Boiling with alc. HBr failed to cleave the lactone ring but isomerized VII into a new lactone, m. 133-4°, [α]<sub>D</sub> - 55.3° (c 6.0 in CHCl<sub>3</sub>). VII was also isolated as a product of the energetic oxidation of II by KMnO<sub>4</sub>. The hypothetical unsatd. ketone expected from the splitting out of H<sub>2</sub>O from III was prepared by heating 0.5 g. III with 1.5 g. Mg(ClO<sub>4</sub>)<sub>2</sub> in 3 cc. toluene for 5 hrs. The cooled reaction mixture was diluted with ether and washed with H<sub>2</sub>O. The evaporation of the dried ether extract and high vacuum distillation of the residual oil

gave 0.4 g. of unsatd. ketone, b<sub>0.4</sub> 130-5°; semicarbazone, C<sub>19</sub>H<sub>33</sub>N<sub>3</sub>O, m. 197-8°. These results, confirming the proposed structure of I, leave undetd. the position of the 2 Me groups split out on dehydrogenation.

IT 93158-29-3, 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl-  
(preparation of)

RN 93158-29-3 CAPLUS

CN 1-Naphthaleneacetic acid, decahydro-2-hydroxy-2,5,5,8a-tetramethyl- (6CI, 7CI) (CA INDEX NAME)



FILE 'CAOLD' ENTERED AT 12:51:44 ON 28 OCT 2004

L5 7 S L3

L5 ANSWER 1 OF 7 CAOLD COPYRIGHT 2004 ACS on STN

AN CA65:20170a CAOLD

TI ferruginol-type diterpenes and proton magnetic resonance characteristics of diterpenic substances

AU McChesney, James D.

TI optical rotation and structure in the labdane series of diterpenoids

AU Carman, R. M.

TI synthesis and stereochemistry of fichtelite-structure and stereochemistry of some reduction products of abietic-type resin acids

AU Marx, John N.

IT 468-68-8	468-81-5	468-82-6	510-98-5	511-01-3	511-02-4
511-03-5	640-28-8	640-29-9	1156-07-6	1235-39-8	1235-40-1
1235-76-3	1235-77-4	1408-33-9	1409-35-4	1412-99-3	1438-55-7
1438-62-6	1438-64-8	1616-86-0	1619-25-6	1757-81-9	1757-83-1
1757-85-3	1757-87-5	1857-24-5	1891-72-1	1908-44-7	1909-92-8
2761-77-5	3650-30-4	3954-67-4	3954-68-5	4176-94-7	4630-08-4
4966-16-9	5956-15-0	5957-33-5	6049-24-7	6138-92-7	6605-72-7
6605-74-9	6605-76-1	6713-91-3	6813-12-3	7292-96-8	10178-31-1

Searcher : Shears 571-272-2528

10/820709

10178-32-2	10207-80-4	10266-75-8	10266-76-9	10266-77-0	10266-78-1
10266-80-5	10266-81-6	10266-82-7	10266-85-0	10266-89-4	10266-91-8
10266-96-3	10267-02-4	10267-04-6	10267-05-7	10267-06-8	10267-07-9
10267-08-0	10267-09-1	10267-10-4	10267-11-5	10267-12-6	10267-14-8
10267-15-9	10267-18-2	10267-19-3	10267-21-7	10267-22-8	10267-23-9
10267-24-0	10267-25-1	10267-26-2	10267-27-3	10267-28-4	10267-29-5
10267-30-8	10267-31-9	10267-32-0	10267-33-1	10267-34-2	10267-35-3
10267-36-4	10267-37-5	10267-38-6	10267-39-7	10305-15-4	10305-16-5
10314-36-0	10314-38-2	10314-40-6	10314-41-7	10314-42-8	10314-43-9
10314-44-0	10314-45-1	10314-46-2	10314-47-3	10314-48-4	10314-50-8
<b>10314-52-0</b>	<b>10314-53-1</b>	10314-56-4	10314-57-5		
10314-59-7	10314-61-1	10314-62-2	10314-63-3	10314-69-9	10314-71-3
10314-72-4	10314-74-6	10314-75-7	10314-76-8	10314-77-9	10314-79-1
10314-80-4	10314-83-7	10314-84-8	10314-85-9	10314-86-0	10314-87-1
10314-95-1	10380-71-9	10395-31-0	10395-32-1	10395-33-2	10395-35-4
10395-36-5	10395-37-6	10395-38-7	10395-39-8	10395-41-2	10395-42-3
10395-43-4	10470-23-2	10470-24-3	10470-25-4	10470-31-2	10483-51-9
11015-78-4	13013-31-5	13346-05-9	13346-07-1	13383-62-5	13384-28-6
13384-46-8	13384-96-8	<b>13456-36-5</b>	13902-83-5	13902-98-2	
14022-42-5	16633-28-6	17829-02-6	17904-64-2	19533-83-6	20404-55-1
22343-28-8	23963-10-2	24460-84-2	25490-89-5	25671-16-3	28644-60-2
33762-81-1	36052-45-6	53771-91-8	55881-96-4	93158-10-2	
<b>93158-29-3</b>	<b>93813-28-6</b>	96749-49-4	97017-02-2		
99831-26-2	99831-27-3	100028-48-6	100194-79-4	100194-80-7	100194-81-8
100232-35-7	100624-45-1	101296-75-7	102216-44-4	102444-58-6	103425-20-3
103476-93-3	106196-13-8	106196-16-1	106300-22-5	106631-38-3	107928-45-0

L5 ANSWER 2 OF 7 CAOLD COPYRIGHT 2004 ACS on STN  
AN CA60:431c CAOLD  
TI bicycloalicyclic compds., resolution of racemates of  
PA CIBA Ltd.  
DT Patent  
TI resolution of racemates of bicycloalicyclic compds.  
AU Prelog, Vladimir  
DT Patent

PATENT NO.	KIND	DATE
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PI	CH 366530			
IT	4242-00-6	20007-72-1	<b>93158-29-3</b>	94997-53-2 95427-55-7

L5 ANSWER 3 OF 7 CAOLD COPYRIGHT 2004 ACS on STN  
AN CA58:1435b CAOLD  
TI two stage oxidation of sclareol  
PA Reynolds, R. J., Tobacco Co.  
DT Patent  
TI two-stage oxidation of sclareol  
AU Schumacher, Joseph N.; Henley, W. M.; Bernasek, E.; Teague, C. E., Jr.  
DT Patent

PATENT NO.	KIND	DATE
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PI	US 3050532	1962
IT	17904-64-2	<b>93158-29-3</b>

L5 ANSWER 4 OF 7 CAOLD COPYRIGHT 2004 ACS on STN  
AN CA54:2679a CAOLD  
TI improving the flavor of tobacco



10/820709

AU Schumacher, Joseph N.

DT Patent

PATENT NO.	KIND	DATE
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PI	US 2905576	1959
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DE 1209473

GB 847201

GB 847201

IT	1216-84-8	3738-00-9	55881-96-4	93158-29-3
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L5 ANSWER 5 OF 7 CAOLD COPYRIGHT 2004 ACS on STN

AN CA53:18093b CAOLD

TI C $\beta$ -C $\gamma$  cleavage of a  $\gamma$ -hydroxy acid by electrolytic oxidation

AU Corey, Elias J.; Sauers, R. R.

IT 50767-77-6 93158-29-3 101082-99-9 101433-46-9 101442-74-4

102945-76-6 102945-77-7 109727-79-9

L5 ANSWER 6 OF 7 CAOLD COPYRIGHT 2004 ACS on STN

AN CA52:4580h CAOLD

TI C $\beta$ -C  $\gamma$  cleavage of a  $\gamma$ -hydroxy acid by electrolytic oxidation

AU Corey, Elias J.; Sauers, R. R.; Swann, S., Jr.

IT 50767-77-6 93158-29-3 94259-75-3 101082-99-9 101433-46-9

101442-74-4 102945-76-6 102945-77-7

L5 ANSWER 7 OF 7 CAOLD COPYRIGHT 2004 ACS on STN

AN CA52:1110h CAOLD

TI total synthesis of pentacyclosqualene

AU Corey, Elias J.; Sauers, R. R.

IT 464-91-5 1216-84-8 56105-46-5 109313-16-8 109727-79-9

111562-36-8 111589-12-9 111589-13-0 116126-47-7

FILE 'USPATFULL' ENTERED AT 12:52:05 ON 28 OCT 2004

L6 5 S L3

L6 ANSWER 1 OF 5 USPATFULL on STN

ACCESSION NUMBER: 2004:248357 USPATFULL

TITLE: Process for the optical resolution of a precursor of sclareolide

INVENTOR(S): Huboux, Alexandre, Pringy, FRANCE

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 2004192960	A1	20040930
APPLICATION INFO.:	US 2004-820709	A1	20040409 (10)

	NUMBER	DATE
PRIORITY INFORMATION:	WO 2002-IB3055	20020731
DOCUMENT TYPE:	Utility	
FILE SEGMENT:	APPLICATION	
LEGAL REPRESENTATIVE:	WINSTON & STRAWN, PATENT DEPARTMENT, 1400 L STREET, N.W., WASHINGTON, DC, 20005-3502	
NUMBER OF CLAIMS:	12	
EXEMPLARY CLAIM:	1	
LINE COUNT:	386	

Searcher : Shears 571-272-2528

10/820709

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB The present invention relates to the field of organic synthesis and more particularly to a new process for the optical resolution of a precursor of sclareolide. This process includes the reaction of [(1RS,2RS,4aSR,8aSR)-2-hydroxy-2,5,5,8a-tetramethyldecahydronaphthalen-1-yl]acetic acid, or an alkaline salt thereof, with an enantiomer of the 2-(methylamino)-1-phenyl-1-propanol, or an ammonium salt thereof respectively, which is used as resolving agent.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L6 ANSWER 2 OF 5 USPATFULL on STN

ACCESSION NUMBER: 96:51024 USPATFULL

TITLE: Process for the production of sclareolide

INVENTOR(S): Schneider, Markus, Duisburg, Germany, Federal Republic of

Stalberg, Theo, Monheim, Germany, Federal Republic of

Gerke, Thomas, Neuss, Germany, Federal Republic of

PATENT ASSIGNEE(S): Henkel Kommanditgesellschaft auf Aktien, Duesseldorf, Germany, Federal Republic of (non-U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 5525728		19960611
	WO 9321174		19931028
APPLICATION INFO.:	US 1994-318790		19941017 (8)
	WO 1993-EP874		19930408
			19941017 PCT 371 date
			19941017 PCT 102(e) date

	NUMBER	DATE
PRIORITY INFORMATION:	DE 1992-4212731	19920416
DOCUMENT TYPE:	Utility	
FILE SEGMENT:	Granted	
PRIMARY EXAMINER:	Evans, Joseph E.	
LEGAL REPRESENTATIVE:	Jaeschke, Wayne C., Drach, John E., Millson, Jr., Henry E.	
NUMBER OF CLAIMS:	20	
EXEMPLARY CLAIM:	1	
LINE COUNT:	292	

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB A process for the production of sclareolide comprising the steps of: (1) providing an aqueous composition comprised of: (a) water; (b) sclareol, abienol, or a mixture of sclareol and abienol, (c) an effective amount of a ruthenium catalyst; and, (d) an emulsifying agent; (2) forming an aqueous alkaline composition by adding an alkali metal hydroxide to said aqueous composition; (3) reacting said aqueous alkaline composition with an oxidizing agent to form a crude product; and

either: (4) further reacting said crude product with base to form the salt of 8 $\alpha$ -hydroxy-11-carboxyl-12, 13, 14, 15, 16-pentanorlabdane and; (5) reacting said salt with acid to form sclareolide;

or: (4) heating said crude product to form sclareolide.

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CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L6 ANSWER 3 OF 5 USPATFULL on STN

ACCESSION NUMBER: 94:80132 USPATFULL

TITLE: Process for producing ( $\pm$ )-2,5,5,8a-tetramethyl-1-(carboxymethyl)-2-hydroxydecalin

INVENTOR(S): Asanuma, Goro, Kurashiki, Japan

Tamai, Yoshin, Shibata, Japan

PATENT ASSIGNEE(S): Kuraray Co., Ltd., Kurashiki, Japan (non-U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 5347048		19940913
APPLICATION INFO.:	US 1993-116605		19930907 (8)
RELATED APPLN. INFO.:	Division of Ser. No. US 1992-995978, filed on 23 Dec 1992, now patented, Pat. No. US 5290955		

	NUMBER	DATE
PRIORITY INFORMATION:	JP 1991-358777	19911229
	JP 1991-358778	19911229
DOCUMENT TYPE:	Utility	
FILE SEGMENT:	Granted	
PRIMARY EXAMINER:	Shippen, Michael L.	
LEGAL REPRESENTATIVE:	Oblon, Spivak, McClelland, Maier & Neustadt	
NUMBER OF CLAIMS:	18	
EXEMPLARY CLAIM:	1	
LINE COUNT:	928	

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB A process for producing ( $\pm$ )-2,2,2,5a-tetramethyl-1-(carboxymethyl)-2-hydroxydecalin, comprising the steps of allowing a carbonate of dihydro- $\beta$ -vinyl-ionol to react with carbon monoxide in the presence of a palladium catalyst to form  $\beta$ -monocyclohomofarnesic acid, cyclizing said  $\beta$ -monocyclohomofarnesic acid in the presence of an acid catalyst to form ( $\pm$ )-norambreinolid, and hydrolyzing said ( $\pm$ )-norambreinolide to form ( $\pm$ )-2,5,5,8a-tetramethyl-1-(carboxymethyl)-2-hydroxydecalin.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L6 ANSWER 4 OF 5 USPATFULL on STN

ACCESSION NUMBER: 94:18193 USPATFULL

TITLE: Process for producing L-ambrox

INVENTOR(S): Asanuma, Goro, Kurashiki, Japan

Tamai, Yoshin, Shibata, Japan

PATENT ASSIGNEE(S): Kuraray Co., Ltd., Kurashiki, Japan (non-U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 5290955		19940301
APPLICATION INFO.:	US 1992-995978		19921223 (7)

NUMBER	DATE
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Searcher : Shears 571-272-2528

10/820709

PRIORITY INFORMATION: JP 1991-358777 19911229  
JP 1991-358778 19911229  
DOCUMENT TYPE: Utility  
FILE SEGMENT: Granted  
PRIMARY EXAMINER: Dentz, Bernard  
LEGAL REPRESENTATIVE: Oblon, Spivak, McClelland, Maier & Neustadt  
NUMBER OF CLAIMS: 4  
EXEMPLARY CLAIM: 1,3  
LINE COUNT: 924

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB (-)-2,5,5,8a-Tetramethyl-1-(carboxymethyl)-2-hydroxydecalin is subjected to lactonization by dehydration to form decahydro-3a,6,6,9a-tetramethyl(3a $\alpha$ ,5a $\beta$ ,9a $\alpha$ ,9b $\beta$ )-(+) -naphtho[2,1-b]furan-2(1H)-one, which is then reduced with a metal hydride to convert it into (-)-2,5,5,8a-tetramethyl-1-(carboxymethyl)-2-hydroxydecalin, followed by dehydrative cyclization to give L-ambrox.

The (-)-2,5,5,8a-tetramethyl-1-(carboxymethyl)-2-hydroxydecalin is produced from its racemic mixture. The resolution is performed using a 1-(aryl)ethylamine. The starting material for the synthesis is beta-ionone.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L6 ANSWER 5 OF 5 USPATFULL on STN

ACCESSION NUMBER: 93:78945 USPATFULL  
TITLE: Process for the production of sclareolide  
INVENTOR(S): Gerke, Thomas, Neuss, Germany, Federal Republic of  
Bruns, Klaus, Krefeld-Traar, Germany, Federal Republic of  
PATENT ASSIGNEE(S): Henkel Kommanditgesellschaft auf Aktien, Duesseldorf, Germany, Federal Republic of (non-U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 5247100		19930921
	WO 9109852		19910711
APPLICATION INFO.:	US 1992-862560		19920622 (7)
	WO 1990-EP2166		19901213
			19920622 PCT 371 date
			19920622 PCT 102(e) date

	NUMBER	DATE
PRIORITY INFORMATION:	DE 1989-3942358	19891221
DOCUMENT TYPE:	Utility	
FILE SEGMENT:	Granted	
PRIMARY EXAMINER:	Cintins, Marianne M.	
ASSISTANT EXAMINER:	Peabody, John	
LEGAL REPRESENTATIVE:	Szoke, Ernest G., Jaeschke, Wayne C., Millson, Jr., Henry E.	
NUMBER OF CLAIMS:	20	
EXEMPLARY CLAIM:	1	
LINE COUNT:	243	

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB A process for the production of sclareolide from sclareol comprising the

10/820709

steps of

A) oxidatively degrading sclareol to a reaction product which is one or both of the following compounds: ##STR1## using either a hypochlorite salt in the presence of a ruthenium salt or potassium permanganate, and

B) oxidizing the above reaction product with a peracid or salt thereof to form sclareolide.

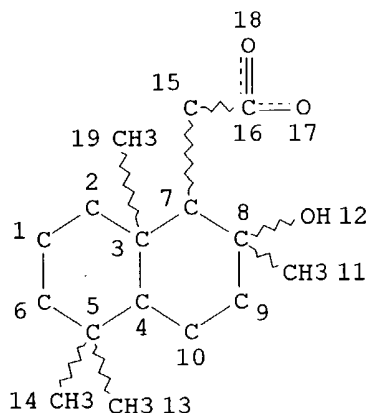
The above process results in good yields and much shorter reaction times than prior art processes.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

(FILE 'CASREACT' ENTERED AT 12:53:34 ON 28 OCT 2004)

L1

STR



NODE ATTRIBUTES:

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RSPEC I

NUMBER OF NODES IS 19

STEREO ATTRIBUTES: NONE

L10 5 SEA FILE=CASREACT SSS FUL L1 ( 11 REACTIONS)

100.0% DONE 1843 VERIFIED 11 HIT RXNS

5 DOCS

SEARCH TIME: 00.00.01

L10 ANSWER 1 OF 5 CASREACT COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 140:164046 CASREACT

TITLE: A process for the optical resolution of a precursor of sclareolide

INVENTOR(S): Huboux, Alexandre

PATENT ASSIGNEE(S): Firmenich SA, Switz.

SOURCE: PCT Int. Appl., 15 pp.

CODEN: PIXXD2

DOCUMENT TYPE:

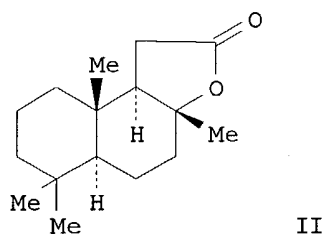
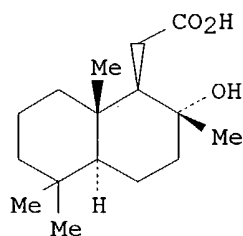
Patent

Searcher : Shears 571-272-2528

10/820709

LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

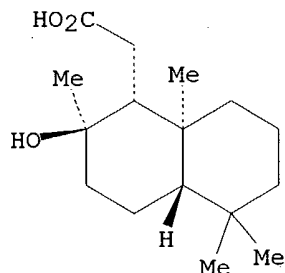
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004013069	A1	20040212	WO 2003-IB2933	20030724
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
US 2004192960	A1	20040930	US 2004-820709	20040409
PRIORITY APPLN. INFO.: GI			WO 2002-IB3055	20020731



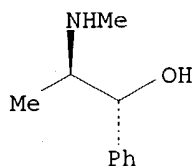
AB The present invention relates to the field of organic synthesis and more particularly to a new process for the optical resolution of a precursor of sclareolide. Said process is characterized by the reaction of [(1R,2R,4aS,8aS)-2-hydroxy-2,5,5,8a-tetramethyldecahydronaphthalen-1-yl]acetic acid (I), or an alkaline salt thereof, with an enantiomer of the 2-(methylamino)-1-phenyl-1-propanol, or an ammonium salt thereof resp., which is used as resolving agent. Thus, I was treated with (1R,2R)-pseudoephedrine in THF to form the diastereomeric salt of (1R,2R,4aS,8aS)-I with (1R,2R)-pseudoephedrine. The diastereomeric salt was treated with 10% aqueous H<sub>2</sub>SO<sub>4</sub> in toluene and the toluene phase containing (1R,2R,4aS,8aS)-I was subsequently treated with acetic acid to give (+)-sclareolide (II) in 91% yield and >98% ee.

RX(1) OF 3      A + B ==> C...

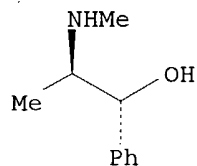
10/820709



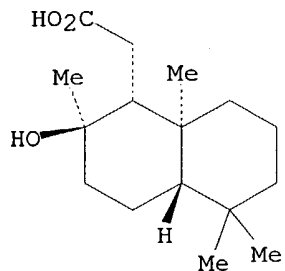
A



B



C: CM 1  
YIELD 92%



C: CM 2  
YIELD 92%

RX(1) RCT A 151123-71-6, B 321-97-1  
PRO C 654076-05-8  
SOL 109-99-9 THF

L10 ANSWER 2 OF 5 CASREACT COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 120:54746 CASREACT  
TITLE: Process for producing L-ambrox  
INVENTOR(S): Asanuma, Goro; Tamai, Yoshin  
PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan  
SOURCE: Eur. Pat. Appl., 18 pp.  
CODEN: EPXXDW  
DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

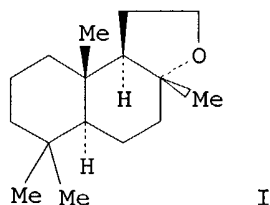
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 550889	A1	19930714	EP 1992-121945	19921223
EP 550889	B1	19960918		
R: BE, CH, DE, FR, GB, IT, LI, NL				
JP 05186388	A2	19930727	JP 1991-358777	19911229

Searcher : Shears 571-272-2528

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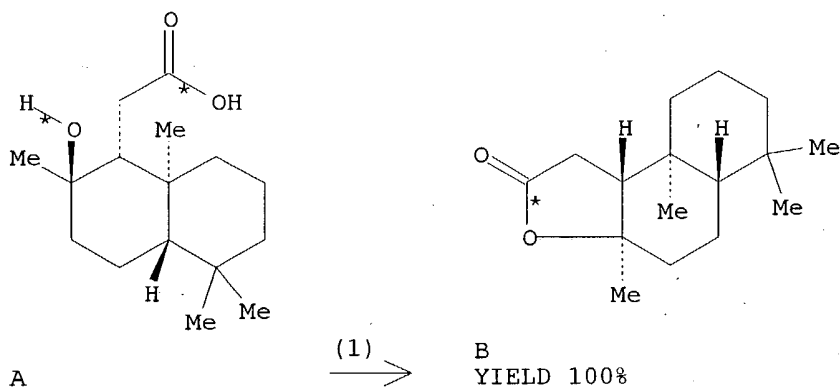
JP 3028874	B2	20000404		
JP 05186452	A2	19930727	JP 1991-358778	19911229
US 5290955	A	19940301	US 1992-995978	19921223
US 5347048	A	19940913	US 1993-116605	19930907
PRIORITY APPLN. INFO.:			JP 1991-358777	19911229
			JP 1991-358778	19911229
			US 1992-995978	19921223

GI



AB Title compound (I) a known perfumery substance is produced with a high optical purity, at a low cost and in an industrial scale by subjecting (-)-2,5,5,8a-tetramethyl-1-(carboxymethyl)-2-hydroxydecalin (II) to lactonization by dehydration, reducing the lactonized compound to (-)-2,5,5,8a-tetramethyl-1-(hydroxyethyl)-2-hydroxydecalin (III) followed by dehydrative cyclization to give I.  $\beta$ -Ionone was reduced to dihydro- $\beta$ -ionone which in 6 steps was converted to II. II was lactonized and reduced to III followed by dehydrative cyclization with p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>Cl to I.

RX(1) OF 6      A ==> B...



RX(1)      RCT    A **13456-36-5**  
              PRO    B 564-20-5  
              SOL    108-88-3 PhMe

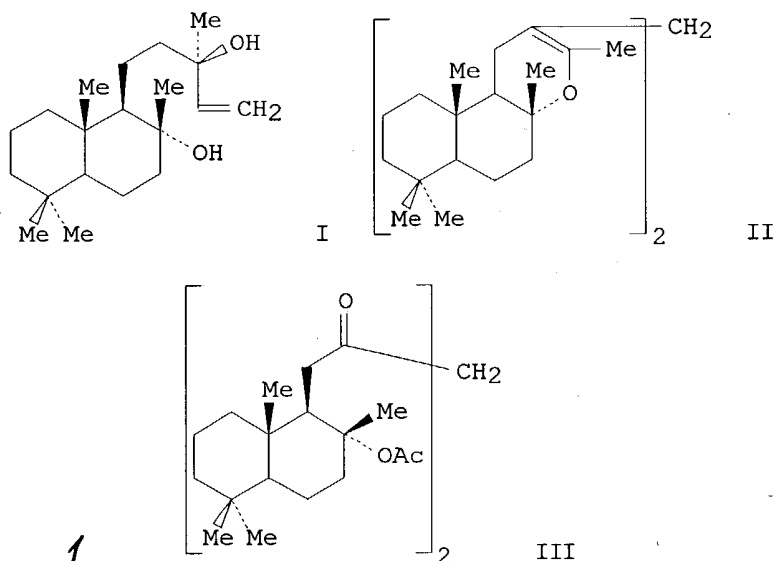
L10 ANSWER 3 OF 5 CASREACT COPYRIGHT 2004 ACS on STN

Searcher :      Shears      571-272-2528



10/820709

ACCESSION NUMBER: 116:129310 CASREACT  
TITLE: Synthesis of bicyclohomofarnesane derivatives of  
bis(8 $\alpha$ ,13-epoxy-14,15-bisnorlabd-12-en-12-yl)methane, a product of sclareol ozonolysis  
AUTHOR(S): Aryku, A. N.; Koltza, M. N.; Vlad, P. F.; Kukovinets,  
O. S.; Odinokov, V. N.; Tolstikov, G. A.  
CORPORATE SOURCE: Inst. Khim., Kishinev, USSR  
SOURCE: Khimiya Prirodnkh Soedinenii (1991), (3), 343-9  
CODEN: KPSUAR; ISSN: 0023-1150  
DOCUMENT TYPE: Journal  
LANGUAGE: Russian  
GI



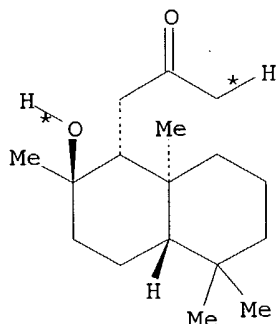
AB A method for obtaining novel fragrant analogs of ambroxide, with a strong amber odor, from Sclareol (I) is described. The key steps in the synthesis of these products are ozonolytic decomposition of I with the formation of bis(8 $\alpha$ ,13-epoxy-14,15-bisnorlabd-12-en-12-yl)methane (II), and its ozonization to bis(13,14,15,16-tetranorlabdan-8 $\alpha$ -acetoxy-12-on-12-yl)methane (III) followed by base-catalyzed decomposition

RX(3) OF 15 ...F ==> H + I...

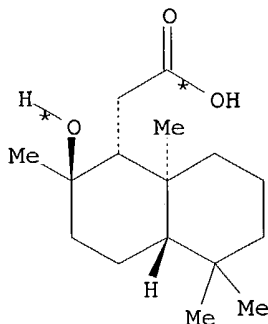
\* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT \*

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(3) →



H  
YIELD 46%



I  
YIELD 48%

RX(3) RCT F 139259-59-9  
RGT J 1310-73-2 NaOH  
PRO H 19895-03-5, I 13456-36-5  
SOL 64-17-5 EtOH  
NTE key step

L10 ANSWER 4 OF 5 CASREACT COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 110:193147 CASREACT  
TITLE: Method of producing 8 $\alpha$ -hydroxy-13,14,15,16-tetranor-12-labdanic acid lactone  
INVENTOR(S): Vlad, P. F.; Kyl'chik, A. N.; Koltza, M. N.; Odinokov, V. N.; Kukovinets, O. S.; Tolstikov, G. A.  
PATENT ASSIGNEE(S): Institute of Chemistry, Academy of Sciences, Moldavian S.S.R., USSR; Bashkir Institute of Chemistry U.S.S.R. From: Otkrytiya, Izobret. 1988, (26), 94.  
SOURCE: CODEN: URXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Russian  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
SU 1409631	A1	19880715	SU 1986-4001468	19860103
PRIORITY APPLN. INFO.:			SU 1986-4001468	19860103

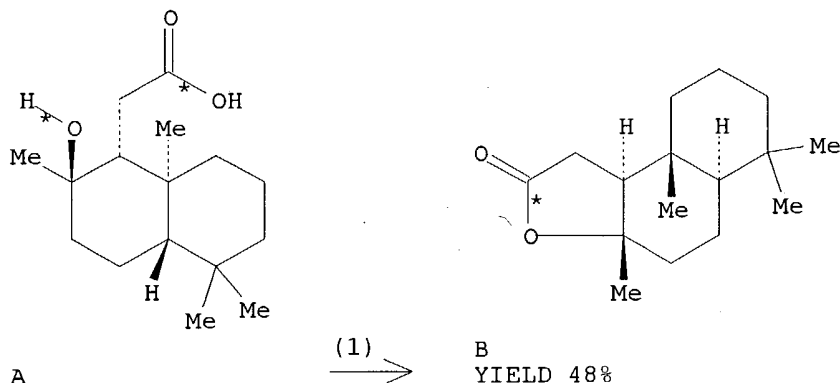
AB The title compound is prepared by oxidation of sclareol. Ecol. purity of the

process is ensured and its selectivity is increased by using O<sub>3</sub> as the oxidizing agent and by reacting at 5-10° with subsequent ozonization of the resulting bis(8 $\alpha$ -13-epoxy-15,14-bis-norlabd-12-en-12-yl)-methane at (+23)°-(-65)°, alkaline cleavage of the resulting product at reflux, acidifying, separation of the acid part, and lactonization at 120-140°.

RX(1) OF 1 A ==> B

Searcher : Shears 571-272-2528

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RX(1) RCT A 151123-71-6  
PRO B 79768-41-5

L10 ANSWER 5 OF 5 CASREACT COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER:

53:100021 CASREACT

TITLE:

CB-Cy Cleavage of a  $\gamma$ -hydroxy acid by electrolytic oxidation

AUTHOR(S):

Corey, E. J.; Sauers, R. R.

CORPORATE SOURCE:

Univ. of Illinois, Urbana

SOURCE:

Journal of the American Chemical Society (1959), 81, 1743-5

CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE:

Journal

LANGUAGE:

Unavailable

GI 1 For diagram(s), see printed CA Issue.

AB cf. preceding abstract A novel elimination process, which has been observed during anodic reaction of a  $\gamma$ -HO acid, is described. The vinyl ketone obtained by the electrolysis in MeOH of I (R = Me, R' = OH) (as NH<sub>4</sub> salt) (cf. preceding abstract) was identified as 4-(1,3,3-trimethyl-1-vinyl-2-cyclohexyl)-2-butanone (II), b0.4 95-6°, n<sub>D</sub>20 1.4857, [ $\alpha$ ]<sub>D</sub>20 -10.4° (c 1.06); semicarbazone, m. 175.5-8.5°, plates from aqueous EtOH. II, also obtained in the electrolysis of the NH<sub>4</sub> salt of I (R = OH, R' = Me), b0.3 93-100°, n<sub>D</sub>20 1.4834. Br (0.8 cc.) added to 1.8 g. NaOH in 14 cc. H<sub>2</sub>O, a 2.9-cc. portion added to 0.204 g. II in 14 cc. H<sub>2</sub>O, stirred 12 hrs. at room temperature, heated 15 min. on

the steam bath, poured into 25 cc. H<sub>2</sub>O, extracted with Et<sub>2</sub>O, and the aqueous phase

acidified with dilute HCl and extracted with Et<sub>2</sub>O gave 0.150 g.

3-(1,3,3-trimethyl-1-vinyl-2-cyclohexyl)propionic acid;

benzylisothiuronium salt, plates, m. 143-5° (aqueous EtOH). II (0.432

g.) in 10 cc. MeOH hydrogenated over 39 mg. 5% Pd-C, evaporated in vacuo, diluted with H<sub>2</sub>O, and extracted with Et<sub>2</sub>O, the extract worked up, and the

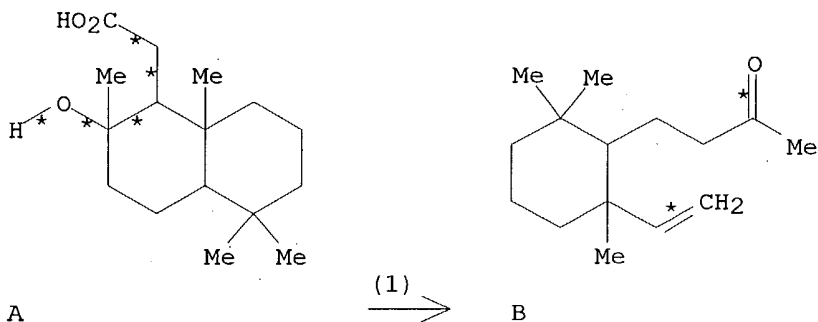
residual

oil chromatographed on Al<sub>2</sub>O<sub>3</sub> gave 0.286 g. dihydro-II, clear oil;

semicarbazone, m. 156.5-8.5°.

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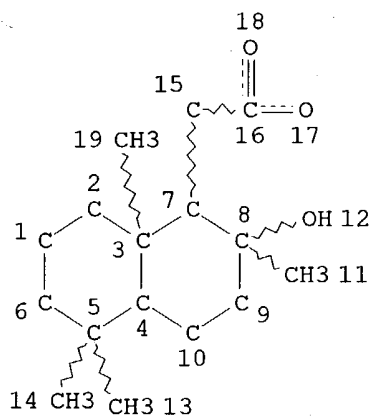
RX(1) OF 1      A ==> B



RX(1)      RCT    A 93158-29-3  
             PRO    B 50767-77-6  
             NTE    Classification: Elimination; Fragmentation; Decarboxylation;  
                     Ring cleavage; # Comments: 30% cyclohexene

(FILE 'DJSMDs, CHEMINFORMRX' ENTERED AT 12:55:14 ON 28 OCT 2004)

L1                STR



NODE ATTRIBUTES:  
DEFAULT MLEVEL IS ATOM  
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:  
RSPEC I  
NUMBER OF NODES IS 19

STEREO ATTRIBUTES: NONE  
L11                0 SEA L1

FILE 'HOME' ENTERED AT 12:55:40 ON 28 OCT 2004